

Research/Technical Note

Comparison of the Effect of Saliva on the Mechanical Properties and Microscopic Profile of Cad/Cam and Conventional Resins: Experimental Study

Asmaa Hatim^{1, 2, *}, Mory Keita^{1, 2}, Hourya Ghazi^{1, 2}, Barry Alpha Amadou^{1, 2}, Fatima Mohaddib³, Hassan Hannach⁴

¹Department of Removable Prosthodontics, Mohammed VI University of Sciences and Health, Casablanca, Morocco

²Dental Sciences Research Laboratory, Mohammed VI University of Sciences and Health, Casablanca, Morocco

³Private Practitioner, Casablanca, Morocco

⁴Department of Materials Science, Energy, and Nanotechnology, Mohammed VI Polytechnic University, Benguerir, Morocco

Abstract

Introduction: For a long time now, the majority of conventional complete denture bases have been made from acrylic resins. Conventional PMMA-based acrylic resins have remained firmly established in our practice because of their many qualities. Dentistry's entry into the era of computer-assisted design and manufacture (CAD/CAM) has offered prosthetic solutions adapted to a wide range of situations and has led to gains in precision, reproducibility and time. This work is an experimental study that investigates the microscopic structure and mechanical behaviour of two groups of acrylic resin intended for the manufacture of total dentures, while focusing on the salivary action on both types. **Materials & Methods:** The experiment was carried out in 3 successive stages: The first stage involved analysing the samples under a scanning electron microscope and evaluating their mechanical behaviour in terms of hardness and tensile strength. The second stage consisted of soaking the samples in artificial saliva according to a protocol described in the literature. The third stage involved carrying out the same types of analysis (SEM and mechanical) on the soaked samples. **Results:** Scanning electron microscope images of conventional PMMA were marked by the presence of black dots of different intensities and diameters ranging from 1 to 10 μm . They are of considerable number and occupy a large surface area in the observed sample. The image revealed spots of high opacity in places. The images were clustered and the domains are randomly oriented. The CAD/CAM images were characterised by the presence of a few black dots of varying intensities, most of which were no more than 3 μm in diameter. Other scattered dots show high opacity with band-like texturing. In the mechanical tensile test, we can say that PMMA - Conventional has better elongation and better plastic deformation, and is therefore better able to absorb external stresses, due to its high elongation capacity. On the other hand, the mechanical strength is lower than that of PMMA - CAD/CAM. The latter has higher mechanical strength but low elongation. **Conclusion:** This study highlights the potential benefits of CAD/CAM resins in terms of microscopic quality and mechanical strength, suggesting their advantageous use in dental prosthetics. However, long-term clinical studies are needed to validate their clinical suitability and durability.

*Correspondence: Asmaa Hatim (ahatim@um6ss.ma)

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Keywords

PMMA CAD-CAM, PMMA-conventional, Microscopy, Mechanical Properties

1. Introduction

For a long time, the majority of conventional complete denture bases have been made from acrylic resins. Acrylic resins based on conventional PMMA have remained firmly established in our practice because of their many qualities (ease of processing, physical, mechanical and aesthetic properties, cost, low toxicity, ease of use, etc.). However, they also have a number of limitations (more sessions, lack of precision, poor stability, etc.) [1-3]. Since dentistry entered the digital era thanks to the work of François Duret in the 1970s, CAD/CAM (computer-aided design and manufacture) has continued to evolve and become more widely available. CAD/CAM offers prosthetic solutions adapted to a wide range of indications. Thanks to the use of a special resin (PMMA CAD/CAM), it offers greater precision, reproducibility and faster turnaround times [4, 5].

Saliva, chewing and food can contribute to the degradation of acrylic resins. An immersed cured resin absorbs water due to the polar properties of the resin molecules, leading to diffusion of free monomers and other products [6]. Salivary enzymes degrade polymers, leading to PMMA degradation [7]. Acids react chemically with the acrylic resin, filling the spaces between the polymer chains and causing the polymer chains to separate [8].

However, one factor that is often overlooked when assessing the performance of these materials is the effect of biological fluids, particularly saliva, on their mechanical properties and durability. Due to its complex chemical composition, saliva can influence the structural stability, wear resistance and biocompatibility of dental materials. This interaction is all the more important in the case of dental materials, which are in constant contact with the oral cavity. Studies examining the influence of saliva on denture base resins are limited.

The aim of this experimental study was therefore to investigate the microscopic structure and mechanical behaviour of two groups of acrylic resin (used in the manufacture of total dentures) in relation to salivary action.

2. Material and Methods

We have established and followed a rigorous work protocol.

1) Sample preparation

We prepared samples of PMMA resin (Poly Methyl Acrylic Methacrylates) for use in the manufacture of total dentures.

2) Manufacturing processes

a) Manufacture of PMMA specimens using CAD/CAM

A brand of CAD/CAM PMMA billet was used in this study: Ava Dent PMMA billets.

Four billets (98 mm diameter × 25 mm thickness) of the same brand were required to mill the PMMA-CFAO specimen. This billet was produced by CAD/CAM injection moulding at a pressure of 3,000 daN.

A 10 cm × 5 cm × 2 mm Plexiglas specimen was sprayed with a contrast spray, stabilised on a custom-made stone base, and mounted in the Ceramillmap 400 CAD/CAM scanner. The digital image of the scanned specimen was processed using Ceramillmind software.

The PMMA billet was milled according to the template provided using the TIZIAN Cut 5 Smart Plus open CAD/CAM milling system.

The milled specimen was then cut from the billet and finished using acrylic tungsten carbide burs and silicon carbide papers. The specimen was then polished using acrylic rubber burs and pumice stone.

Both surfaces were polished. Specimen dimensions were checked using a digital caliper. The specimen was stored in distilled water for 48 hours to remove residual monomer.

b) Conventional fabrication of heat-cured PMMA specimens

The conventional PMMA specimen measuring 10 cm × 5 cm × 2 mm was coated with two coats of petroleum jelly, then placed in a vacuum-mixed Type III dental stone and a 50/50 vacuum-mixed dental stone. Bottling was carried out in a two-part mould using a Hanau Varsity bottle.

The flask was opened and the PMMA model removed. The rectangular cavity surfaces were sealed with two layers of Cold Mold putty. The heat-cured acrylic resin powder and liquid were proportioned and mixed according to the manufacturer's instructions. The paste was pressure packed and cured using a short cure cycle in a thermostatically controlled water bath.

Excess acrylic was trimmed and the specimen was finished and polished exactly as was done for the PMMA - CAD/CAM specimen. Specimen dimensions were checked using a caliper. Similarly, the specimen was stored in distilled water for 48 hours to minimise residual monomers.

3) Standardisation of samples

We prepared (Figure 1):

- a) Plates made from resin fired in a water bath, known as conventional resin
- b) CAD/CAM resin plates
- c) Dimensions: 10 cm × 5 cm × 2 mm

However, to ensure that the sample analysis surfaces were naturally prepared and that the cutting planes were not systematic, we used liquid nitrogen to explode the samples and then use the fragments for microscopic and mechanical analysis [9].

Liquid nitrogen is a transparent liquid that boils at a temperature below 196 °C at atmospheric pressure. Under normal conditions, liquid nitrogen is therefore colder than the temperature mentioned.

This property makes it a widely used cryogenic fluid in biology for cooling and freezing living tissue. Such a cold liquid evaporates rapidly at room temperature.

To preserve it, it is kept in heat-insulated containers (the coffee thermos principle). It can be used for a multitude of experiments in its different states.

The experiment we tried with liquid nitrogen in our study involved immersing a solid in it.

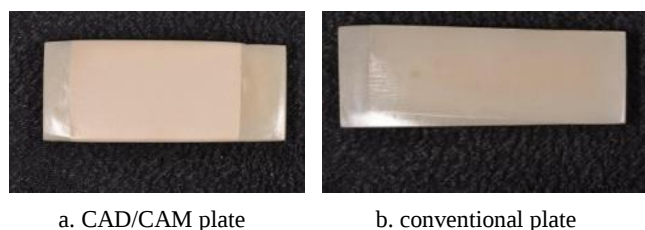


Figure 1. PMMA samples.

4) Experimental protocol

The experiment was carried out in collaboration with the Polymers and Critical Phenomena Physics Laboratory (LPPPC) of the Ben M'sik Faculty of Science at Hassan II University in Casablanca and the Materials Science and Nano-engineering Laboratory at Mohammed VI Polytechnic University in Bengurir.

The experiment took place in 3 successive stages:

Analysis of initial samples before soaking SEM analysis of samples

The SEM is capable of producing high-resolution images of the surface of samples using the principle of electron/matter interactions.

It consists of a beam of electrons scanning the surface of the sample to be analysed, which in response re-emits certain particles. These particles are analysed by various detectors, enabling a three-dimensional image of the surface to be reconstructed.

Insulating samples (biological samples, polymers, etc.) must also be metallised, i.e. coated with a thin layer of carbon or gold. However, the thickness of this metallic layer will prevent the detection of very small details. A lower-energy electron beam can therefore be used to avoid charging the sample (and therefore losing visibility), and the metallic layer will no longer be necessary.

Synthetic replicas can be made to avoid the use of original

samples when they are not suitable or available for SEM examination due to methodological obstacles or legal problems.

The samples obtained are observed using an M.E.B. (Philips® XL 30) at 20 keV in backscattered electron mode in order to observe the nature and molecular composition of the different types of resin. Images at different magnifications were taken after the carbon fibre samples had been metallised. Secondary electron mode is used to observe the surface condition of the samples. Finally, microscopic observation of the polished samples was carried out in 4 views of 4 samples, giving a total of 16 views (Figure 2).



Figure 2. SEM analysis of samples.

Mechanical analysis of samples

In this study, we also chose to study the mechanical properties of the initial samples, namely:

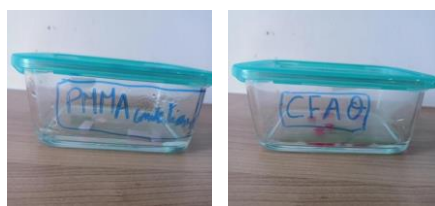
1. **Tensile strength:** The tensile test is a physical experiment that provides information on the elastic behaviour and degree of resistance to fracture of a material when subjected to uniaxial stress.
2. **Elongation:** Elongation or extension is the variation in length of a material under tension and the range of this elongation. Its capacity for plastic elongation is called ductility.
3. **Fracture limit:** Fracture mechanics tends to define a material property that can be expressed in terms of its resistance to brittle (fracture) or ductile (elongation) fracture.
4. **Hardness:** The hardness of a material is its capacity to offer a certain resistance to the penetration of a defined object. Depending on the resistance obtained, it will then be possible to define its hardness [10-12].

Soaking the samples in artificial saliva

We then soaked the samples in an ARTISIAL-type artificial saliva solution for 21 days.

This artificial saliva was composed of potassium chloride, sodium chloride, magnesium chloride, calcium chloride, dipotassium phosphate and mono-potassium phosphate [8].

The two types of resin were stored separately in two glass jars containing 50 cm³ of ARTISIAL-type artificial saliva at room temperature (21 °C) in the absence of light (Figure 3).



a. Conventional PMMA b. PMMA - CAD/CAM

Figure 3. Soaking of PMMA samples in artificial saliva.

Microscopic and mechanical analysis of quenched samples

After this period, the quenched samples were re-examined for the same microscopic and mechanical analyses.

3. Results

SEM observation results

Analysis of PMMA before soaking in artificial saliva

We took images of sections at progressive magnifications. We were thus able to use 3 images at 3 scales of 20 μm , 10 μm and 4 μm (Figure 4).

The Spectrum overlay associated with the images shows the technical details of the observation, in particular the direct observation time of 48.6 seconds, the resolution of 131.7 eV and the exposure in kV.

The margin of error in the observation is given for 3 samples in the associated table. The results used are for the sample with the lowest risk of error, which is 5.17%. (Figure 4).

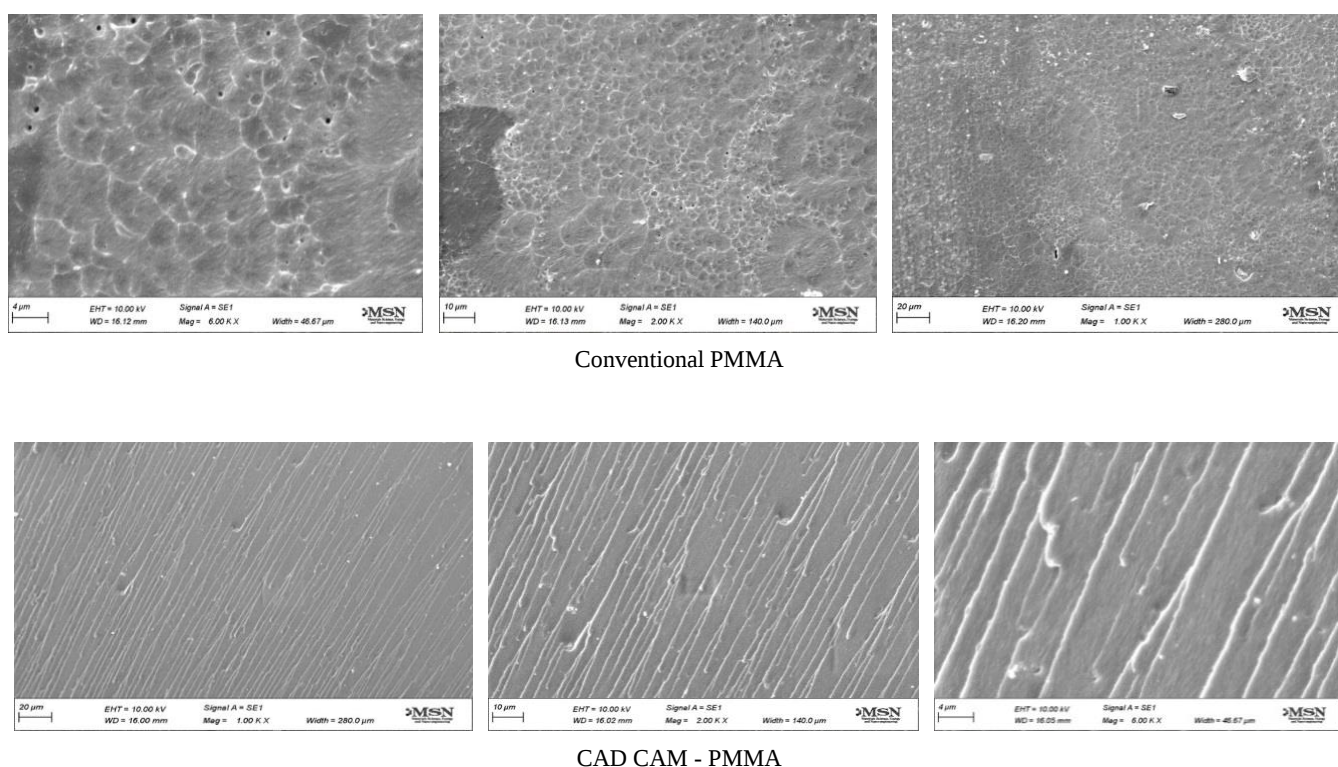


Figure 4. SEM appearance of PMMA.

Image interpretation:

In conventional mode:

1. Presence of black dots of different intensities. They vary in diameter from 1 to 10 μm . They are numerous and occupy a large area of the observed sample.
2. Dots of high opacity in places.
3. Images of clusters - grains
4. Randomly oriented domains

In CAD/CAM mode:

1. Presence of a few black dots of varying intensity, most

of which are no more than 3 μm in diameter.

2. Presence of a few highly opaque dots. They are scattered.
3. The orientation is oblique.
4. Texturing: Band structure

Analysis of PMMA after soaking in artificial saliva (Figure

5)

No significant difference in SEM observation before and after soaking in artificial saliva (ARTISAL), for both samples.

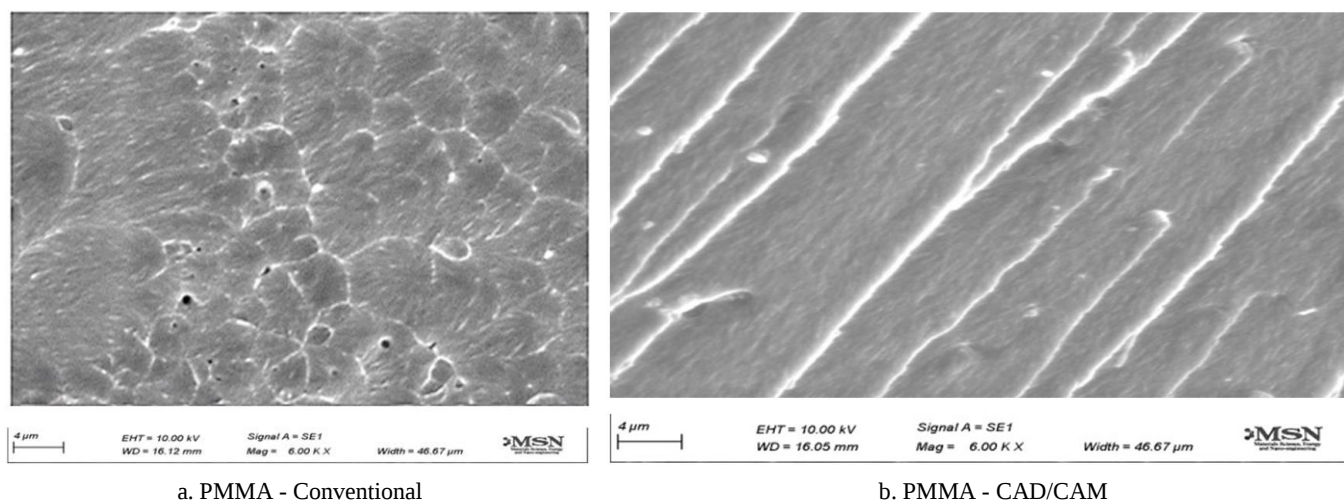


Figure 5. SEM appearance after dipping in ARTISAL.

Results of the mechanical analysis of the samples (Table 1)

The tensile test enabled us to follow the evolution of the elongation of the materials as a function of the force applied.

In this way, the materials can no longer continue to elongate and at a given moment pass from the plastic zone to the deformation zone and then to fracture.

Table 1. Comparison of tensile test results on PMMA - Conventional and PMMA - CAD/CAM before and after dipping in ARTISAL.

Propriétés mécaniques	Conventional PMMA		CAD CAM PMMA	
	Before	After	Before	After
Maximum force (N)	636,72	408,61	294,10	461,79
Extension (mm)	8,63	9,52	6,14	9,16
Tensile strength (MPa)	39,60	11,83	24,21	13,37
Breakdown time (S)	28	27,6	18,8	29

We found that for an almost equal amount of time (28 seconds) of application of mechanical stress: Conventional PMMA before immersion in ARTISAL is significantly higher than that applied after immersion and that for CAD/CAM PMMA after immersion is higher than that before (Figure 6).

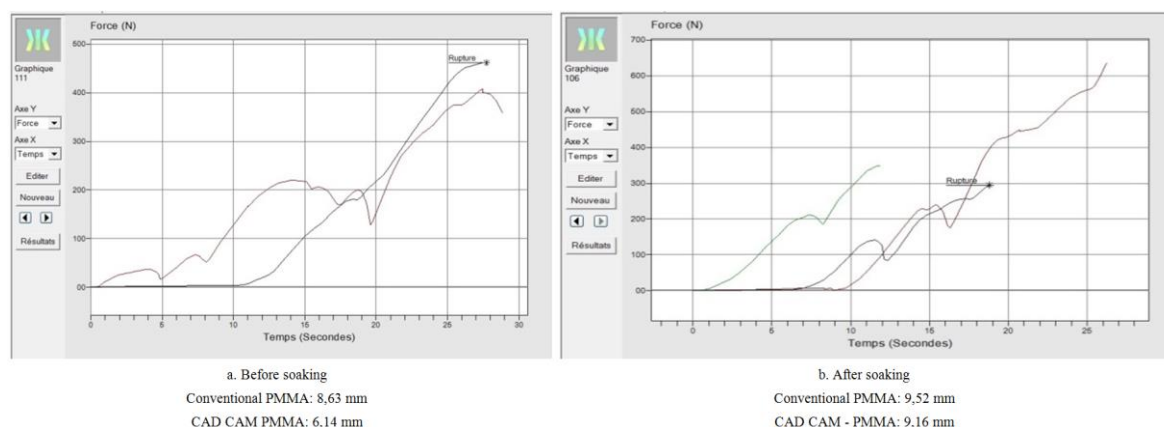


Figure 6. Mechanical behaviour of PMMA-Conventional and PMMA-CAD in tensile tests.

The elongation values for PMMA/CFAO are lower than those for conventional PMMA before and after soaking in artificial saliva.

4. Discussion

Acrylic resin remains the material of choice for the manufacture of bases for removable dental prostheses [13].

These synthetic macromolecular resins belong to the large family of plastics and offer advantageous properties thanks to the use of a mixture of polymers or copolymers. These polymers improve mechanical and thermal resistance on the one hand, and reduce water absorption on the other [5].

In our study, PMMA samples produced using conventional and CAD/CAM techniques were studied from two angles:

1) Scanning electron microscope (SEM) observation

To carry out these observations, our samples had to be cut in such a way that they were compatible with the sizes and dimensions recommended for each apparatus used. For this, we chose to split the samples using liquid nitrogen by bursting, to avoid having striations at the level of the fascias of rupture and to obtain a real cut. On the other hand, if we had made a mechanical cut, impurities would have been present and we would have had to go through a polishing phase.

Scanning electron microscope analysis of the samples before soaking in saliva showed dark areas or pores, which means that there is no signal detected in the presence of a vacuum. These pores are abundant in the PMMA - Conventional sample and rare in the PMMA - CAD/CAM sample. The latter, on the other hand, shows white light intensities in places, meaning that the surface is well oriented towards the secondary electron detector.

This analysis also showed that PMMA - Conventional has an appearance of randomly oriented clusters or grains in the mass. However, PMMA - CAD/CAM has a textured appearance in the form of parallel, well-oriented bands. This can be explained by the fact that it is made up layer by layer, and these layers are thin. This to-and-fro process during the application of the layers gives the material time to breathe, whereas with the conventional method, the PMMA is condensed after it has been deposited as a whole mass in one go. During this condensation, gas is released from the surface, creating preferential paths in the internal structure of the material.

After soaking, although scanning electron microscopy revealed no noticeable changes before and after soaking in artificial saliva, for both types of sample. However, we cannot deduce that saliva has no effect on the resin, given that our results are primary and our sample is small.

Other studies [14], on the other hand, have shown that certain concentrations of elements in the materials differ slightly from the technical data supplied by the manufacturer:

- a) The microstructure is finer after immersion in artificial saliva.

- b) Little degradation of the elements making up the material observed.
- c) The particle size distribution is different and slightly coarser.
- d) Particle morphology is similar.
- e) The presence of holes between the grains and the matrix in the SEM images of the material before and after soaking.

Other studies have also revealed a different SEM ultrastructure after exposure of CAD/CAM resin to saliva [15].

2) Mechanical study: tensile test

With regard to the mechanical study carried out, we can say that PMMA-Conventional exhibits better elongation and better plastic deformation, indicating a greater ability to absorb mechanical stress. However, the mechanical strength is lower than that of PMMA-CFAO. The latter has higher mechanical strength but low elongation.

Other studies have concluded that CAD-CAM complete removable prostheses offer a number of improved mechanical and surface properties and are not inferior to those noted for conventional prostheses. This may justify the effective use of this type of material in clinical practice [16, 17].

The most remarkable fact about the results obtained in the study by Terese Palacios et al. On the impact of saliva on PMMA - CAD/CAM is that there are consistent trends that allow us to state that immersion in saliva significantly modifies the mechanical behaviour of the material in all the aspects analysed. Reductions in properties of more than 30% (as in the case of fracture toughness) can be caused, which can seriously compromise their long-term viability [14].

What we noticed during our literature search was that few studies are dedicated to comparing the mechanical behaviour of PMMA-Conventional and PMMA-CFAO in terms of tensile strength, elongation and hardness.

The aim of our study, like others studying this polymer (PMMA), is to supplement and enrich the scientific literature on the mechanical behaviour of this material in tensile tests, using both conventional and digital (CAD CAM) manufacturing techniques. The aim is to improve our prosthetic treatments for the edentulous population, especially the geriatric population.

3) Limitations and prospects of the study

Our study presents several incomplete aspects and in order to arrive at scientifically valid conclusions, further tests and observations must be carried out.

The tensile tests carried out must be optimised by increasing the reliability of the tensile conditions in order to analyse the elastic and plastic behaviour of each grade.

Fracture modes can be analysed at the level of fracture surfaces to improve mechanical properties, particularly fracture resistance, with the aim of increasing the viability of complete dental prostheses.

Material thickness can also influence fatigue strength. In fatigue, it is difficult to control crack propagation.

To do this, fractography is used to show the morphology of the cracks or breaks and their starting points or initiation zones.

To finalise our study and exploit it scientifically, we need to investigate the link between the mechanical properties of PMMA - whether conventional or CAD/CAM - and their microscopic structures.

We therefore intend to carry out other complementary characterisation tests, in particular RAMAN or infra-red scattering spectroscopy.

The tests we plan to carry out involve both samples without and with exposure to saliva, in order to be able to conclude significant results regarding the effect of saliva on the microstructure and mechanical properties of PMMA.

5. Conclusion

This study has highlighted differences in microstructure and mechanical behaviour between conventionally and CAD/CAM designed PMMA samples, as well as the effect of artificial saliva on these parameters.

However, we were able to obtain initial results that need to be confirmed and improved by complementary tests and additional manipulations in order to better understand the long-term impact of saliva on the different types of resin used in adjunctive prosthetics.

The ultimate goal is to improve the characteristics of this material to achieve better clinical results in prosthetics.

Abbreviations

PMMA	Polymethyl Methacrylate
CAD – CAM	Computer-Aided Design - Computer-Aided Manufacturing
SEM	Scanning Electron Microscope

Conflicts of Interest

The authors declare no conflicts of interest.

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