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Effects of saliva on additive manufacturing materials for dentistry applications: Experimental research using flexural strength analysis

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ABSTRACT
Accurate and reliable results in orthodontics heavily depend on selecting the right impression materials. With the rise of digital technology and additive manufacturing techniques, it has become necessary to characterize experimentally the materials used to design prosthetic bases. In this study, the mechanical properties of Polyetheretherketone, Nylon6, Nylon12, and Polypropylene are analyzed, as impression materials commonly used in dentistry applications. Specifically, the effect on their flexural elastic modulus of the exposure to working environment conditions is also investigated by means of 3-point bending test performed on virgin materials and samples immersed in saliva for 72 hours. The proposed approach revealed significant behavior in terms of loss in mechanical performances. These findings have significant implications for the proper selection and use of AM materials in dental applications.

Section: RESEARCH PAPER

Keywords: additive manufacturing; dentistry; flexural strength; polymers; 3D printing

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1. INTRODUCTION

In orthodontics, the accurate depicting of a patient's dentition is essential to properly identify and diagnose malocclusions, to plan out the course of action, to aid with assessments, and to track the effectiveness of medical treatments [1]. For this purpose, in the dentistry field, impression materials are typically used to accurately replicate the shape of the oral structures (including teeth), creating a negative form to generate the cast mold [1]. In the early days of dentistry, materials known as impression compounds were commonly used to make casts of the oral cavity [1]. However, these materials were rigid and did not allow for the creation of accurate casts that could reproduce all the tissues of the oral cavity [2]. As a result, there was a growing need for materials that could remain elastic even after setting. In the mid-20th century, the use of hydrocolloids became widespread due to their ability to provide the required elasticity and to enable the creation of accurate casts of undercuts [3]. Over time, other materials, such as PVS have been introduced, which exhibit less shrinkage over time compared to hydrocolloids, and these advancements have greatly improved the accuracy and reliability of dental impressions [4]. In the late 1990s, the most used impression materials in dentistry became addition reaction silicone, polyether, and reversible hydrocolloid [4], [5]. The ideal impression material should possess certain characteristics that are suitable for use in the clinical environment. These materials must be adaptable to the oral structures, be resistant to tearing, and be easily removable without causing trauma to the surrounding tissues [3]. The material's physical and mechanical properties must also be carefully considered to ensure suitability for the clinical application and can provide optimal precision and marginal adaptation [3], [4], [6]. Moreover, the ability of the material to flow and adapt to the tooth structure is crucial for the creation of a quality restoration. Therefore, the impression material must possess good fluidity and moisture tolerance to effectively slip and imprint itself into the tooth structure that needs to be copied [3]. In fact, the marginal precision of an in-vitro dental restoration is typically 50 μm [4]. In summary, the selection of the appropriate impression material is essential to achieving accurate and reliable results in dentistry. Although impressions are widely used in dentistry due to their simplicity, ease of use, and cost-effectiveness, during the mixing stage, air bubbles may be incorporated, leading to inaccuracies in the model. Furthermore, the typical impression process can be messy, and traces of material may remain on the patient's teeth, necessitating removal [2]. Despite the development of new impression materials, it remains challenging to eliminate the possibility of human errors during each stage of the process [7]. Additionally, the high fabrication time can impact the chairside or intra-operative period. Plaster casts, which are commonly made from impressions, can be damaged and require storage space. Patients may also need multiple models to observe the progress of treatment [7]. Overall, while impressions have several advantages, they also have limitations and drawbacks that must be considered. Because of this, the development of new materials and techniques continues to improve the accuracy and efficiency of the manufacturing, but careful attention must be paid to all stages of the process to achieve optimal results. With the advent of new technologies, it is now possible to obtain a 3D representation of oral structures and tissues using high-resolution and high-fidelity scanners. This technology enables the storage of 3D files in digital format, providing the opportunity to reuse them when necessary [3], [4]. This alternative approach offers several advantages over traditional methods, including reduced risk of infection, improved patient comfort, and shorter chair-time. Additionally, displaying the 3D images to patients during pre-treatment can simplify the therapy explanation and allow for greater patient involvement in the decision-making process [7].

Intraoral scanners capture jaw and tooth geometries in digital format, which can then be processed using computer-aided design (CAD) software. This software enables manipulation of the 3D data and facilitates the development of the final product through computer-aided manufacturing (CAM). In the dental-technical field, CAD/CAM technology has the potential to bypass laboratory experience and allow for the fabrication of dental restorations directly at the chairside. This approach offers significant time savings and can result in one-appointment dental restorations [8]. In dentistry, there are two main CAD/CAM techniques: Subtractive Manufacturing (MM) and Additive Manufacturing (AM). MM involves removing material from a block to produce a prosthetic, typically using metal such as Cobalt-Chromium (Co-Cr), Zirconia (ZrO2), Titanium (Ti), or polymers such as methyl methacrylate (PMMA) or Polyetheretherketone (PEEK) [9]. This method provides the highest accuracy in prosthesis production and is commonly used in dentistry. AM, on the other hand, is an industrial process that involves the production by adding layer-upon-layer based on a 3D model. This method allows for faster production and is suitable for highly customized manufacturing, with the ability to create irregular grooves, crannies, valleys, and bone-like morphology. As a result, AM is ideal for individual dentistry implant [6], [10]. However, due to the inferior flexural strength of printed prostheses compared to MM, AM is typically suggested for interim crown and fixed partial dentures to avoid extended mastication periods [9]. Studies have shown that AM can produce durable dental frameworks and can reduce material waste by up to 40 % compared to MM [10]. Additionally, up to 98 % of the waste can be reused in future manufacturing processes, thereby reducing environmental impact [10]. Furthermore, compared to milling technology, 3D printing offers a wider range of usable materials and more flexible available machines, making it an attractive technology for new applications in the dentistry field [11], [12]. Of all the available techniques, the two most preferred additive manufacturing technologies in dentistry are Fused Deposition Modelling (FDM) and Selective Laser Sintering (SLS) [12]. However, the use of metals in dental implants is not recommended [13]. Despite their exceptional mechanical properties, the biocompatibility of metals is a known limitation. Furthermore, materials such as stainless-steel exhibit poor corrosion resistance in comparison to titanium, which can lead to allergic reactions. In addition to biocompatibility, aesthetic appearance is an important consideration in dental implant design [14]. For these reasons, polymers have emerged as a promising alternative to metals in this regard, offering improved aesthetics. To further enhance the properties of polymers, ceramic and fiber reinforcements have been incorporated into polymer matrices, resulting in polymer matrix composites. Compared to metals, polymer composites have a significantly lower weight, which is one of their major advantages [14]. The elastic modulus of metals is higher than that of bones, which can lead to stress-shielding and failure if the mechanical load is not well-distributed along the adjacent tooth framework [14]. Therefore, the use of polymer composites in dental implant design has the potential to offer improved biocompatibility, aesthetics, and mechanical properties [14]-[16]. The literature lacks extensive research where the properties of materials, generally used in AM processes, are investigated in working environments for dentistry applications. In fact, saliva in dentistry is often used to estimate the loss of color from an aesthetic point of view, the increase of sample mass, rather than the number of bacteria settling the specimen. The evaluation of the performance in terms of elastic modulus before and after the material's immersion is a new proposal in dentistry field.

This research aims to extend the knowledge on properties and performances of polymers for prosthetic bases in dentistry. By means of flexural strength analysis, this study focuses on the experimental characterization of mechanical properties of four different polymers: Polyetheretherketone (PEEK), Nylon6, Nylon12, and Polypropylene (PP), which samples are obtained by FDM and SLS manufacturing processes. As the digestion phase starts in a secreted-saliva environment, a comparison between flexural modules of materials immersed for 72 hours in an artificial-solution of saliva at an ambient temperature of 25 °C to virgin materials is also presented. On the long term, this work will enable the possibility to create customized 3D-printed prostheses directly in the dental office, improving results in orthodontics filed.

2. MATERIALS AND METHODS

In this research, the properties and the behaviour of PEEK, Nylon6, Nylon12, and PP materials are investigated. PEEK has high processability, and 3D printing makes it suitable for fabricating structures for the oral cavity.

PEEK exhibits remarkable resistance to corrosion and can maintain its mechanical characteristics at temperatures as high as 120 °C. When used in a body fluid environment at 37 °C, PEEK's wear is reduced, thereby minimizing the release of harmful particles, and decreasing the immune response. One of PEEK's main advantages is its Young's modulus, which is similar to that of cortical bone (3-4 GPa) [17]. Additionally, by incorporating other compounds, such as carbon fibers, it is possible to enhance the mechanical properties of PEEK [15].

Nylon6 is a thermoplastic material widely used in various engineering applications, including automotive and textiles, due to its ease of processing and excellent mechanical properties [18]. The addition of fillers can further enhance its modulus and strength, resulting in a composite polymer. Nylon6's biocompatibility can be attributed to the presence of amide groups in its chemical structure, which resemble the chemical structure of natural peptides. This similarity enables cells to disguise themselves from the immune system, thereby suppressing the host's reaction to a foreign body [19].

Nylon12 finds application in various industrial fields. However, compared to Nylon6, it has a lower melting point and mechanical strength. Nevertheless, Nylon12 exhibits excellent flexibility, high pressure resistance, low density, extreme temperature resistance, and thermal stability. Moreover, Nylon12 demonstrates outstanding impermeability, chemical resistance, and stability in humid environments, making it particularly suitable for water distribution systems that require precise dimensional accuracy [19], [20].

Polypropylene is a commercial plastic with a low density and excellent mechanical, physical, and chemical properties. One of the most significant advantages of this material is its high temperature resistance [21]. In the biomedical industry, PP is also used for its hydrophilic characteristics, which promote cell adhesion through the cytokine response [22]. Furthermore, the porosity of PP facilitates interaction between the host and the implant, allowing for earlier formation of capillaries [23].

As a preliminary study, where the focus of the research is the material property itself, a simplified geometry of the test samples is preferred over a complex and more realistic design. The specimens used in this study were realized with dimensions of 85 mm in height, 10 mm in width, and 2 mm in thickness, and they are shown in Figure 1. The samples were provided by ZARE, and the printing settings used in the manufacturing process are not given in this paper.

While the PP samples were realized using SLS printing technique, all the other samples were realized through FDM. The technology behind SLS is based on a laser beam that strikes a powdered material lying in a build plate and heats it to a temperature just below the melting point. Once the first layer is completed, a rake drags a veil of material from the powder chamber to the build plate waiting for the execution of the successive layer, which has the resolution of 60 μm [24], [25]. Even if during the sintering processes the powders remain partially melted resulting in porous internal structures and rough surface finishes, the mechanical properties of SLS products are excellent. On the other hand, the FDM is one of the most used printers in the market due to the low-cost availability. A stepper motor driver let a thermoplastic filament to run across a hot nozzle capable to melt the material at a specific temperature previously set. Usually, the nozzle can move along X, Y and Z axes and allows the material to deposit, creating the desired object layer by layer. Temperature of the print head, speed of the driven stepper motor and the thickness (approximate accuracy 35–40 μm) of the layers are some of the parameters that can affect the printing quality, the printing-time, and the final costs. The surface roughness and the visible graduation layers are drawbacks of this technology [26]-[28].

A 3-point bending flexural strength test was selected for the material characterization, and the flexural modulus is picked for determining material properties degradation due to exposure to saliva. The flexural modulus, or bending modulus, is an intensive property that is obtained as the ratio of stress to strain in flexural deformation (ASTM D790). The flexural modulus is determined from the slope of a stress-strain curve produced by a flexural test, assuming a linear stress strain response. A schematic of a flexural modulus measurement setup is shown in Figure 2.

From Figure 2, the flexural modulus is analytically defined as [28]:

$$E_{\mathrm{f}} = \frac{L^3 F}{4\, w\, d\, h^3}. \quad (1)$$

The experimental research was carried out in the laboratories of the University of Perugia. For this study, a LLOYO LR 30K was used. A pre-load, whose intensity depended on the specific material, a feed rate of 1.7 mm/min, defined in the ISO 178 A, and a maximum deformation of 1 mm were the experimental conditions selected for the experiments (see Figure 3).

During each test, the stress σ and the strain ϵ measurements are collected, and the flexural modulus can be estimated in the region of linear elasticity by:

$$E_{\mathrm{f}} = \frac{\sigma}{\epsilon}. \quad (2)$$

In this study, the flexural modulus is initially determined, by means of the procedure described above, for virgin materials. The second step consists of immersing the samples in an artificial saliva (*Saliva Sintetica CTS*) in an environmental temperature of 25 °C for 72 hours, time required for the fluid absorption by polymer material [29]. In this way, the effect of the first digestion environment on the properties of materials is obtained. After the

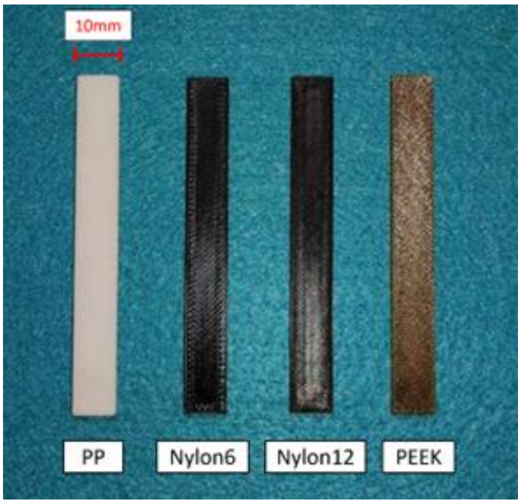

Figure 1. Tested samples materials and geometry.

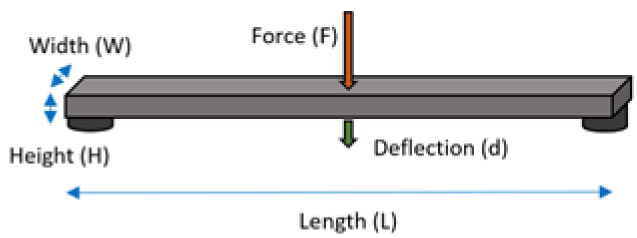

Figure 2. Flexural modulus measurement: 3-point bending test.

Figure 3. Example of PP sample mounted on the LLOYO flexural strength test machinery.

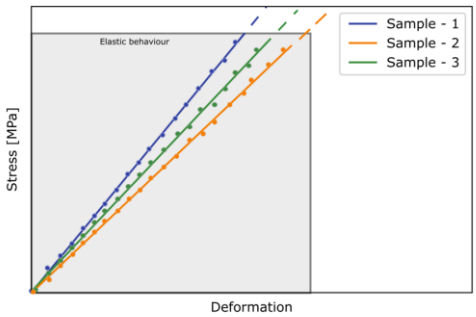

Figure 4. Example of linear interpolation of measured data for a generic material (differences in behavior are enlarged for the sake of comprehensiveness).

curing process, the flexural strength test is repeated, and the flexural modulus of samples exposed to saliva is measured and compared to the one obtained in the virgin condition.

For the sake of repeatability, this procedure is applied to 3 specimens made of the same material, and, considering the 4 materials analysed, a total of 12 samples are tested for this research. Figure 4 shows an example of the results of a flexural strength test measurement for a generic material (differences in behaviour are enlarged for the sake of comprehensiveness).

Solid lines in Figure 4 are obtained as linear interpolation of actual points of measurement (scatter points). From the linear interpolation in the region of elastic behaviour, the flexural elastic modulus is obtained with (2) for each tested sample. In this way, for every material in both virgin and exposed-to-saliva conditions, a mean value of the flexural modulus is given. Also, repeatability analysis was conducted for all tests performed, both on virgin samples and those immersed in saliva.

3. DISCUSSION

An overview of the results obtained as described in Section 2 is given in Table 1. As expected, the results confirm that the exposure to the saliva degrades the properties of the material.

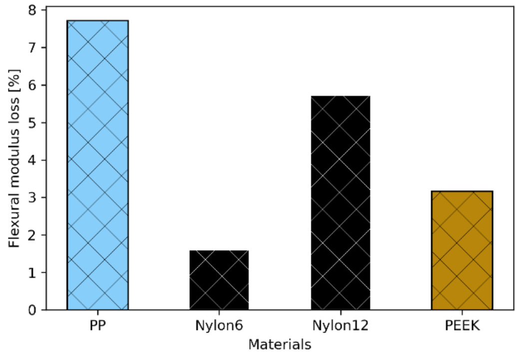

Figure 5. Flexural modulus loss for PP, Nylon6, Nylon12 and PEEK due to saliva exposure.

PEEK shows a better performance in terms of flexural behaviour, with 1794.08 MPa. On the other hand, the Nylon12 shows the lowest values, both from the virgin samples (977.86 MPa) and the samples immersed in saliva (921.83 MPa). Figure 5 shows the flexural modulus loss for the analysed materials.

While Nylon6 shows the smallest loss in mechanical performances with a 2 % loss in flexural modulus, PP has the highest decrease among the specimens. The reason for this behaviour can be related to the AM printing technology. In fact, as the SLS increases the porosity of the sample surface, the ability to absorb liquids also increases [30]. Similarly, PEEK shows a decrease in performance of only 3 %. This is also due to the intrinsic characteristic of the material, which is inherently hydrophobic, with a contact angle of 80-90 degrees [16].

The repeatability analysis, performed with a 2 σ interval at the 95 % of confidence, demonstrate the goodness of the setup and the experiments. In fact, the analysis led to the lowest value of 0.25 %, and a highest of 3.83 %.

The results obtained indicate how saliva has a concrete impact in the elastic modulus performance of each material. On a practical level, this study shows a first approach to the possible development of prostheses with complex geometries, printed with these materials using additive manufacturing technologies. This research can provide the means to predict the performance of these prostheses over time in a reconstruction of the daily work environment.

4. CONCLUSIONS

This paper presents a first attempt to broaden the knowledge of properties of materials used for 3D additive manufacturing in dentistry applications. In this sense, the properties of four materials used prosthetic bases in dentistry were studied in terms of flexural strength modulus: Polyetheretherketone (PEEK), Nylon6, Nylon12, and Polypropylene (PP). Specifically, the

Table 1. Overview of the experimental results.

	Virgin					Saliva				
	Test 1 in MPa	Test 2 in MPa	Test 3 in MPa	Mean in MPa	Repeatability	Test 1 in MPa	Test 2 in MPa	Test 3 in MPa	Mean in MPa	Repeatability
Nylon6	1360.93	1354.78	1365.72	1327.14 ± 43.50	3.79 %	1282.22	1318.96	1316.30	1305.83 ± 16.72	1.48 %
Nylon12	1003.62	963.43	966.51	977.85 ± 18.26	2.16 %	910.67	905.02	949.78	921.82 ± 19.90	2.49 %
PEEK	1818.47	1767.71	1799.06	1795.08 ± 20.91	1.35 %	1842.56	1742.04	1734.48	1738.26 ± 3.78	0.25 %
PP	1095.33	1070.30	1011.53	1059.05 ± 35.12	3.83 %	988.23	1008.21	935.65	977.36 ± 30.60	3.62 %

effect of the saliva, as main component of the first digestive environment, on their mechanical performances was investigated by determining the flexural modulus loss in the materials. Two different additive techniques were used to manufacture the samples used in the study, and 3-point bending tests were performed for determining flexural elastic moduli. Surface material properties and manufacturing techniques were found to be the main factors impacting the properties of the materials exposed to the saliva for 72 hours. Future developments will try to overcome the limitation of this research, *i.e.,* involving a parametric study based on different times of exposure [29], immersing materials in different acid-fluids [31], studying the color loss after the immersion for appearance considerations [31], and, mostly, using more complex geometries of samples for representing actual dental prosthetic designs

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