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The Study of Some Mechanical Properties of Some Composite Materials with Different Types of Matrices and Reinforcement from Chromat-type Isophthalic Resin Granules NPG

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A B S T R A C T

Using the experimental determinations obtained on the basis of compressive stress, some mechanical properties were studied for composite materials with the matrix of three types of resin, epoxy, unsaturated polyester and hybrid based on Dammar natural resin, which was reinforced with isophthalic resin granules NPG (Neopentyl Glycol) Chromat Kayan / Javari / Payette type. The stress strain diagrams, compressive yield strength, compressive strength and modulus of elasticity in uniaxial compression were obtained. With the EDS analysis, the graphical distribution of the atomic spectra of the elements identified in the hybrid resin was determined and the image of the fracture surface of a hybrid resin specimen was presented based on the stereomicroscopic analysis (SEM).

Keywords: composite materials, compressive yield strength, compressive strength, modulus of elasticity in uniaxial compression, atomic spectrum of elements

1. Introduction

In the applications where the isophthalic entrapment granules NPG (Neopentyl Glycol) were to be used, several processes for their production were developed. An example of such a process can be found in [1], where a process was developed for the preparation of aqueous suspensions of vesiculated cross linked polyester resin granules in which the granules have a maximum drying shrinkage of 5% of their diameter. This process consists in the polymerization of an unsaturated carboxylate polyester with acid values between 5-50 mg KOH/g and an unsaturated ethylenic monomer, through a double emulsion process and in the presence of a base such as oxides, hydroxides or metal salts. Another process for the production of isophthalic resin granules NPG is described in [2] and consists in the manufacture of polyester in several stages, including an immersion stage in a swelling medium that allows the kinetics of the post-condensation stage to be increased in solid phase.

Some properties of isophthalic resin granules NPG have been investigated both in separate studies and in association with other components. For example, in [3] the properties of a surface covered with a composite material imitating granite were studied. The respective composite was made from a matrix based on polyester or acrylic resin (with the related reinforcements), and resin granules with

based on polyester or acrylic resin (with the related reinforcements), and resin granules with thermoplastic properties were used as reinforcement. The thickness of the surface coating was uniform and the appearance obtained was of granite.

Various properties of unsaturated polyesters (resistance to thermal shocks, some elastic properties and water absorption) as well as applications of their use for the manufacture of kitchen sinks and countertops were studied in the paper [4].

In the paper [5], an efficient strategy for the chemical recycling of unsaturated polyester resin (UPR) was developed through the selective cleavage of C-O bonds.

More and more fire safety regulations have led to greater demands being placed on structural thermosets such as epoxy and unsaturated polyester resins. As a result of these requests, flame retardant thermosetting polymers of high ecological performance were developed, using fire retardant strategies with monomers [6].

Another method of polymer fireproofing is the use of aluminium trihydroxide (ATH), which is an ecological and economical additive, but which has a low thermal stability. This issue is critical in determining the fire protection and thermal stability of composites. In the study [7] some results were obtained by which the thermal stability of aluminium trihydroxide was improved by combining it with nano-porous silica known as silica air gel (SA), as a hybrid filler in unsaturated polyester resin.

In the same direction of obtaining mechanical and fire performances of glass fiber reinforced composites, in the work [8] the effects of matrices from co-reinforced mixtures of an unsaturated polyester (UP) with inherently fire-retardant phenolic solutions (PH) were investigated and which forms carbon.

The study of some mechanical properties for composite materials with the orthophthalic unsaturated polyester resin matrix reinforced with isophthalic resin granules NPG of the Chromat Buzzi/Perdido type were also investigated in the paper [9].

Data on some mechanical and chemical properties of Chromat-type isophthalic resin granules NPG (Neopentyl Glycol) can be accessed from the manufacturer's website [10, 11].

In papers [12, 13], some chemical and mechanical properties of hybrid resin with volume proportions between 50 and 80% Dammar natural resin were studied. More specifically, the tensile strength, percentage elongation at break and modulus of elasticity were determined based on the characteristic curves. A variation of these characteristics was recorded as follows: it had values between 19.5-20.9 MPa; had values between 1.89-2.05%; had values between 1630-1810 MPa.

In this paper, a hybrid resin with a volume proportion of 60% natural Dammar resin and 40% epoxy resin was made. Based on the EDS analysis, the graphic distribution of the atomic spectra of the chemical elements identified in the hybrid resin was established, and some mechanical properties of the same resin were studied based on the compression stress.

Also, on the basis of compression stress, by comparison, the mechanical properties of some composite materials were investigated, in which three types of isophthalic NPG resin granules of the Chromat Kayan / Javari / Payette type were used as reinforcement, and separately three types of resins used as matrices, more precisely epoxy resin, unsaturated polyester resin and hybrid resin with 60% Dammar.

As applications, it is recommended to use these composite materials in the field of construction, or to make sanitary objects.

2. Materials and methods

2.1. Preparation of samples

Casting of the bars from which the samples were cut for the compression stress was carried out at a controlled temperature between 21 – 23oC.

To make samples from composite materials, Chromat type NPG isophthalic resin granules were used as reinforcement, and three types of resins were used as matrix:
- Resoltech 1050 epoxy resin with Resoltech 1058S related hardener [14];
- NORSODYNE S 20202-A unsaturated polyester resin [15];
- a hybrid resin based on Dammar natural resin (with a volume proportion of 60% Dammar, and 40% Resoltech 1050 epoxy resin with Resoltech 1058S its related hardener).
All the samples made were submitted to the compression test. The type of materials from which the specimens were made, their weight and abbreviation are presented in Table 1.20 specimens from each set were made and the dimensions of the specimens required for compression were 14.7 mm x 14.7 mm x 14.7 mm.

Table 1. Types of samples made and their characteristics

No.	Materials for making the samples	Test weight [g]	Abbreviation
1.	hybrid resin based on Dammar, in which the volume proportion of natural resin is 60%	3.4	D xx
2.	epoxy resin Resoltech 1050 with the related hardener Resoltech 1058S	3.8	E xx
3.	unsaturated polyester resin - orthophthalic NORSODYNE S 20202 A	3.9	P xx
4.	composite materials with the hybrid resin matrix based on Dammar and the reinforcement from isophthalic resin granules NPG of the Chromat Kayan type	4.0	DK xx
5.	composite materials with the hybrid resin matrix based on Dammar and the reinforcement from isophthalic resin granules NPG of the Chromat Javari type	4.1	DJ xx
6.	composite materials with the hybrid resin matrix based on Dammar and the reinforcement from isophthalic resin granules NPG of the Chromat Payette type	4.2	DP xx
7.	composite materials with the epoxy resin matrix and the reinforcement from isophthalic resin granules NPG of the Chromat Kayan type	4.5	EK xx
8.	composite materials with the epoxy resin matrix and the reinforcement from isophthalic resin granules NPG of the Chromat Javari type	4.6	EJ xx
9.	composite materials with the epoxy resin matrix and the reinforcement from isophthalic resin granules NPG of the Chromat Payette type	4.6	EP xx
10.	composite materials with the matrix of unsaturated - orthophthalic polyester resin and the reinforcement of isophthalic resin granules NPG of the Chromat Kayan type	4.4	PK xx
11.	composite materials with the matrix of unsaturated polyester resin - orthophthalic and the reinforcement of isophthalic resin granules NPG of the Chromat Javari type	4.5	PJ xx
12.	composite materials with the matrix of unsaturated polyester resin-orthophthalic and the reinforcement of isophthalic resin granules NPG of the Chromat Payette type	4.6	PP xx

Figure 1 shows samples of specimens for compression stress, made with a matrix based on Dammar and reinforcement from isophthalic resin granules NPG of the Chromat Kayan / Javari / Payette type.

Figure 1. Specimens of specimens for compression stress with matrix based on Dammar

2.2. Technical characteristics of the equipment

The chemical composition of the hybrid resin was determined based on EDS analysis. This analysis was performed with the scanning electron microscope QUANTA INSPECT F50 [16] equipped with field emission electron gun – FEG (field emission gun), with a resolution of 1.2 mm and energy dispersive X-ray spectrometer (EDS), with a resolution at MnK of 133 eV; EDAX chemical micro composition analyser and its related software for performing local micro-composition analyses.

Since the SEM analysis required a much higher magnification than the EDS microscope, the electron microscope – Hitachi model S3400N/type II [17] was used for this analysis, with the following characteristics: SE resolution: minimum 3.0 nm at 30kV (x100,000, WD = 5mm, high vacuum mode); minimum 10 nm at 3kV (x30,000, WD = 5mm, high vacuum mode); BSE image resolution: minimum 4.0 nm at 30kV (x60,000, WD = 5 mm, low vacuum mode); magnification range between 5x and 300,000x; acceleration voltage: 0.3 kV – 30 kV; electron gun with voltage: in stages with self-field and fixed field with continuously adjustable voltage; beam alignment: two-stage electromagnetic deflector; objective opening: 4 holes with click-stop system with diameter of 30, 50, 80 and 150 microns; image displacement: minimum ± 50 microns at WD = 10 mm.

The LLOYD Instruments Lrx PLU mechanical testing machine [18] with the following characteristics was used for compressive testing: maximum value of the applied force: 2.5 kN; stroke size was between 1 and 735 mm; application speed between 0.1 and 500 mm/min; NEXYGEN analysis software.

3. Results and discussions

Since the mechanical and chemical properties of the two synthetic resins and of the NPG isophthalic resin granules of the Chromat type, have been carefully studied by the manufacturers, a similar study is necessary for the hybrid resin used.

In the first part of the experimental research, the chemical composition was determined for the hybrid resin based on Dammar natural resin (with a volume proportion of 60% Dammar).

In the second part of these experimental determinations, the samples made from the composite materials described in section 2.2. were subjected to compression stress, the stress-strain diagrams being obtained. In order to be able to compare the behaviour of the composite materials made, the diagrams were compared according to the reinforcement used.

3.1. Chemical composition of hybrid resins

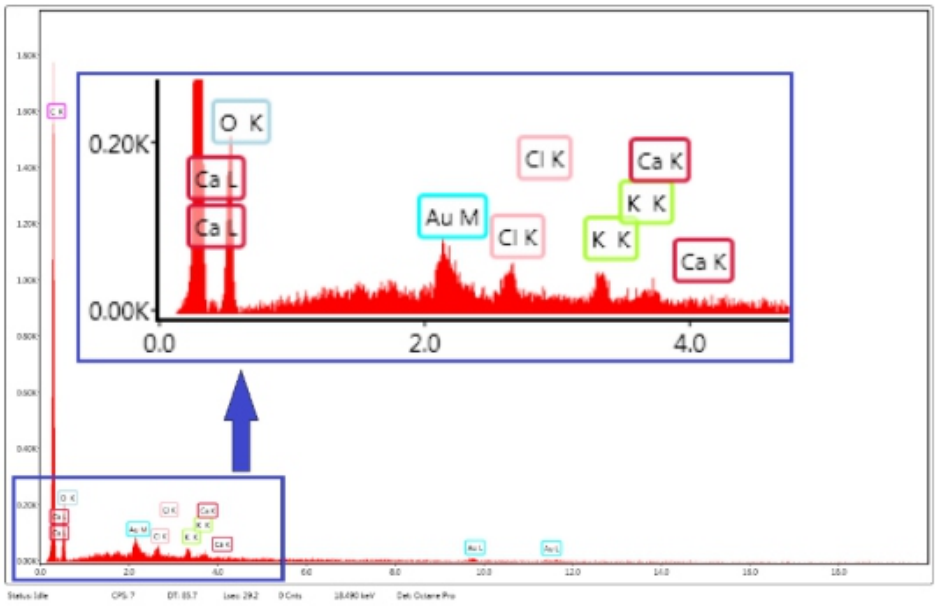
In order to establish the graphical distribution of the atomic spectra of the chemical elements in the hybrid resin, it was necessary to take a sample. The scanning electron microscope cannot record micrographs on this sample because it does not exhibit conductivity. This disadvantage was overcome by preparing the sample before it was subjected to EDS analysis, namely it was inserted for 60 seconds into a metallizer provided with a gold target in which a vacuum was created and a very large amount of little gold The final result of the chemical composition is not influenced by this process because it is only a gold coating to be able to do the analysis (at most, gold is also identified as an element in the EDS analysis).

The sample was analysed with the EDAX detector which gave the graphical representation of the distribution of the atomic spectra of the identified elements and the numerical values of the chemical composition of the hybrid resin.

The investigation of the morphological characteristics related to the sample was carried out both on the surface and in the section.

In Figure 2 this graphical distribution data of the atomic spectra of the elements identified in the hybrid resin. The main area of the graphical distribution of the spectra has been magnified 3.5 times within the

figure in order to be correctly seen.



The spectrum obtained at the intensity of 18,490 keV highlights the presence of the elements carbon, nitrogen, oxygen, chlorine and potassium in the proportion that can be found in Table 2. This structure was obtained based on the EDS analysis and it kept the chemical composition of a hybrid resin sample.

Table 2. Chemical composition of a hybrid resin sample

Element	Weight %	Atomic %	Error %	Net Int.	K Ratio	Z	R	A	F
C K	68.44	74.07	5.08	280.59	0.4669	1.0113	0.9935	0.6745	1
N K	5.85	5.43	32.37	3.07	0.0043	0.9909	1.0035	0.0734	1
O K	24.88	20.22	13.13	44.75	0.0245	0.9731	1.0126	0.1011	1
Cl K	0.28	0.1	23.51	11	0.0024	0.8369	1.0718	1.0247	1.0272
K K	0.55	0.18	13.6	20.3	0.0051	0.835	1.0815	1.0692	1.044

3.2. SEM analysis of the fracture surface of the hybrid resin

Based on the stereomicroscopic analysis (SEM), in Figure 3, the fracture surface image of a representative specimen from the hybrid resin set is presented. This analysis was carried out in compliance with [19].

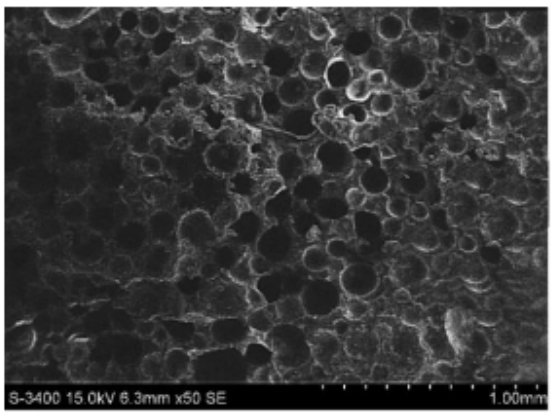


Figure 3. Fracture surface of a representative specimen from the hybrid resin set

3.3. The compression test of the three types of resin used

It must be stated at the outset that this request was made in compliance with [20]. Figure 4 shows the stress-strain diagram obtained after the compression stress of some representative samples from the three sets of resins used.

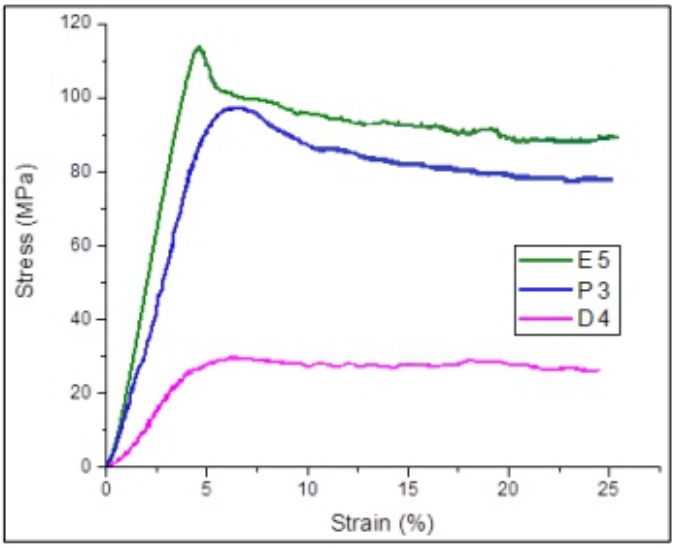


Figure 4. Stress-strain diagram obtained from compression stress a of representative samples from sets E xx, P xx and D xx

3.4. The compression test of the composite materials made

Figure 5 shows the stress-strain diagram obtained after the compression stress of some representative samples from the three sets of composite materials with the reinforcement isophthalic resin granules NPG of the Chromat Kayan type.

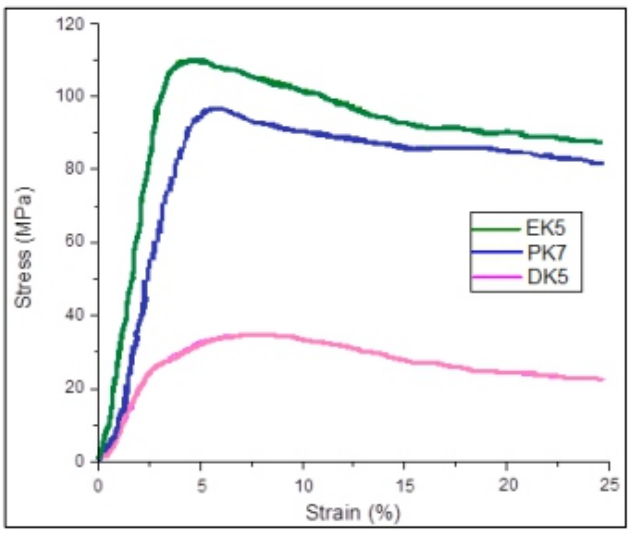


Figure 5. The stress-strain diagram obtained from the compression stress of some representative samples from the EK xx, PK xx and DK xx sets

Figure 6 shows the stress-strain diagram obtained after the compression of some representative samples from the three sets of composite materials with the reinforcement of NPG Chromat Javari isophthalic resin granules.

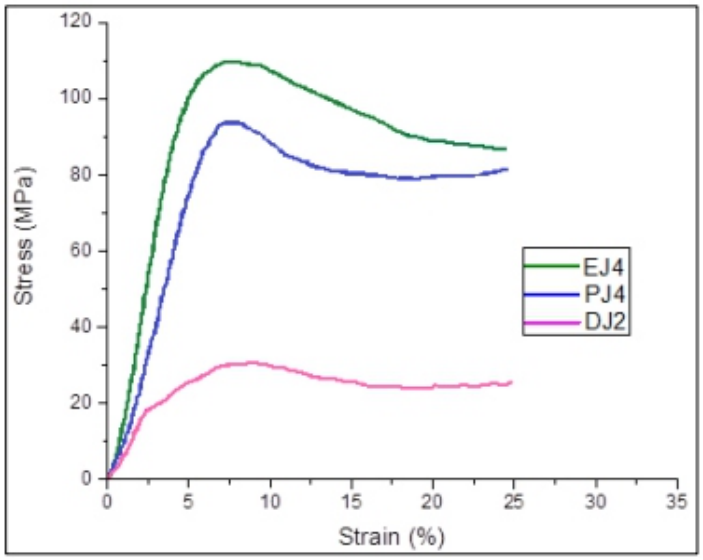


Figure 6. The stress-strain diagram obtained from the compression stress of some representative samples from the sets EJ xx, PJ xx and DJ xx

Figure 7 shows the stress-strain diagram obtained after the compression stress of some representative samples from the three sets of composite materials with the reinforcement of isophthalic resin granules NPG of the Chromat Payette type.

Table 2 shows the averages of the main values of compressive yield strength Rp,0.2[MPa], compressive strength Rc [MPa]and modulus of elasticity in axial compression E[GPa]. The compressive yield strength was determined for the resins and composites studied are materials with a high elongation at break, at which permanent plastic deformations occur.

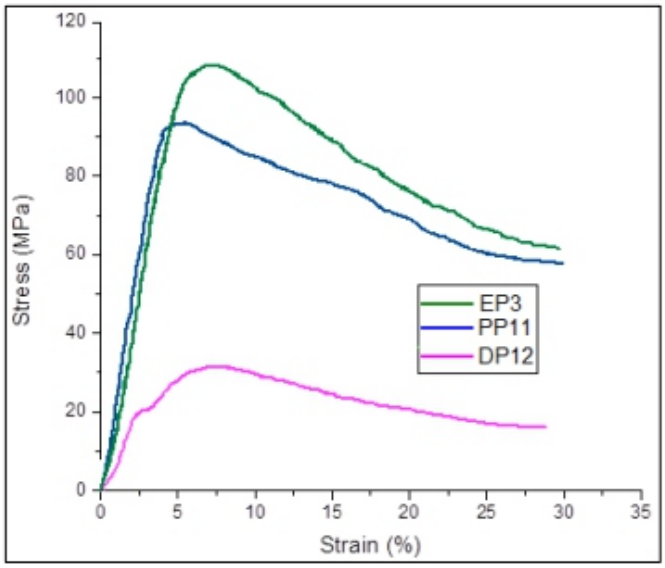


Figure 7. The stress-strain diagram obtained from the compression stress of some representative samples from the sets EP xx, PP xx and DP xx.

The values presented in the following table were obtained as follows: the first two values, the lowest, were eliminated, and for the remaining 8 values, their arithmetic mean was made.

Table 2. Mean values and deviation for the main mechanical characteristics obtained on the basis of compressive stress

Sample	Compression yield strength $R_{p,0.2}\,[MPa]$	Compressive strength $R_c\,[MPa]$	Modulus of elasticity in uniaxial compression $E\,[GPa]$
E	112.02 (±3.03)	113.84 (±3.86)	29.82 (±1.24)
P	94.79 (±2.95)	97.04 (±2.57)	20.27 (±1.16)
D	23.26 (±1.24)	25.14 (±1.08)	6.98 (±0.51)
EJ	100.95 (±3.92)	109.84 (±3.24)	26.04 (±1.97)
PJ	88.93 (±2.74)	92.34 (±2.98)	24.75 (±1.64)
DJ	22.34 (±1.55)	34.93 (±1.92)	10.59 (±0.67)
EK	106.38 (±3.59)	110.75 (±3.85)	33.57 (±1.32)
PK	94.82 (±2.57)	97.84 (±2.74)	22.19 (±1.09)
DK	29.04 (±2.04)	35.95 (±2.18)	10.79 (±0.94)
EP	103.52 (±3.03)	108.02 (±3.18)	28.33 (±1.63)
PP	90.66 (±2.22)	93.71 (±2.84)	29.26 (±1.47)
DP	20.88 (±1.43)	34.21 (±1.46)	11.36 (±0.92)

It can be seen that the mechanical properties obtained for composite materials with hybrid matrix and reinforcement from Chromat-type NPG isophthalic resin granules are superior to those obtained for composite materials with various types of hybrid resins and reinforcements obtained from agricultural waste [21, 22].

The chemical composition and the values of the mechanical properties for the composites with various types of matrices and reinforcements from NPG isophthalic resin granules of the Chromat allow their use in dental technics laboratories. More precisely, in the dental impressions, instead of the special gypsum, the composite material with Chromat granules will be poured. After hardening, the impressions will be removed and the teeth will be polished on which the dental crowns will be made.

An advantage of using these composites instead of special gypsum is that composites are more difficult to fracture when impressions are removed. In addition, fracture of the gypsum leads to impression loss which involves repeating the dental impression and this leads to high final costs.

4. Conclusions

SEM analysis of the surface of the fracture section of a hybrid resin specimen highlights the presence of multiple air bubbles. A possible explanation may be that air bubbles formed as a result of poly merization remain trapped inside the hybrid resin because it polymerizes more slowly than synthetic resins. However, in the case of reinforcing this hybrid resin with various types of fibers / fabrics /

granules, a significant reduction in the number of air bubbles was found in the composite materials made
because the volume proportion of the resin in the composite material is much lower than in the case of the samples made only from resin.
The analysis of the stress-strain diagrams obtained from the compression load shows that the value of the compressive strength of the composite materials reinforced with Chromat-type isophthalic resin granules NPG decreases very little (by approximately) compared to the value of the compressive strength
of the synthetic resin used as a matrix, respectively records. an increase (about) over the compressive strength of the hybrid resin.
The compressive flow limit values and respectively the elastic modulus in uniaxial compression of all the composite materials made decrease compared to the respective values of these characteristics for the constitutive molds. Based on the analysis of the failure sections and the hybrid stress-strain diagrams,
it can be found that the failure of the resin specimens was sudden, without plastic deformation and no flow phenomenon. This type of phenomenon is generally characteristic of brittle materials.
The values determined for the characteristics of these mechanical composite materials allow their use in the field of construction, to please some surfaces (floors, walls), or to make some sanitary objects (sinks, tubs, countertops, etc.), or in the field of dental technics for making molds on which dental crowns
are to be made.

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Biomaterials Used in the Prosthetics of Facial Fractures

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ABSTRACT

The etiology of maxillofacial injuries is represented by car accidents, human aggression, and work-related trauma. Trauma to the maxillofacial area requires a complex treatment that involves functional and aesthetic rehabilitation. To restore the functionality of the damaged areas, a correct surgical technique is necessary along with the use of the ideal prostheses for a better reconstruction. Along with the development and improvement of facial fractures osteosynthesis techniques, an attempt was made to identify the right material to complete these surgical methods. We analyze in this paperwork the benefits of polyetheretherketone-based materials in the prosthetics of different facial fractures in comparison with other materials like titanium.

Keywords: polyetheretherketone, titanium, maxillofacial osteosynthesis

1. Introduction

A complete history of biomaterials has not yet been written, but their multi-millenary development can Biomedical alloys became essential parts of modern medical applications [1]. Facial fractures are difficult to manage. It is necessary to identify the ideal material for the reduction of fracture to be harmonious with the most favorable aesthetic and functional results.

Trauma injuries of the face are commonly encountered in emergency medicine. More than half of patients with these injuries have multiple traumas that require coordinated management between emergency physicians and surgical specialists in oral and maxillofacial surgery, otolaryngology, plastic surgery, ophthalmology, and trauma surgery [2]. Important sensory systems are contained within the face (e.g., vision, auditory, somatic sensation, gustatory, olfaction, and vestibular system) and also, vital structures, of the head and neck are intimately associated (airway, blood vessels, nerves, and digestive tracts. From the anatomical point of view, the face is made up of vertical and horizontal buttresses where the bone is thicker to increase the resistance forces of this bone structure. Reduction and fixation of these key areas are the basis of maxillofacial rehabilitation [2]. Fracture fixation surgery, like minimally invasive plate osteosynthesis, shifted in the direction of saving more tissue and toward smaller dissection. The plating technique started to be used for different bones, however, a broader clinical adoption must be considered after refined conclusions.

Osteosynthesis with miniplates and screws is a technique that brings a series of advantages both to the surgical treatment of fractures and to the functional and physiognomic rehabilitation of the patient.

This technique achieves rapid primary healing of the fracture site, and effective stabilization of the bone fragments, eliminating the need for immobilization and thus reducing the patient's discomfort. Also,

thanks to the direct visual control, the technique allows a precise anatomical reduction of the fracture.
Along with the development and improvement of facial fracture reconstruction techniques, an attempt was made to identify the right material to complete these surgical methods.
A lot of opportunities and challenges there are in the creation and characterization of biomedical alloys. Features of these materials have been designed to be in contact with blood, as replacements for soft and hard tissues [3].
However, they cannot satisfy the increasing requirements for large-scale production owing to the degradation of metals. Physical surface modification could be an effective way to enhance their bio functionality [1].
Continuous research and development of the polymer industry with applications in all medical fields have their ground in the importance of these biomaterials in the health domain [4].
The most used biomaterials in bone prosthetics are by far titanium, polyetheretherketone (PEEK), and a new representative with multiple potentials - BioHPP. Titanium is frequently used in facial implantation based on its durability, low weight, and biocompatibility which offers it good osseo integration. As part of the polyetheretherketone family of polymers, polyetheretherketone is a very efficient bioinert material, which makes it proper for implantation in the human body. Compared to a titanium-based material used to restore joints, polyetheretherketone is more elastic and mirrors the human bone. Research conducted in this area proved the benefits of BioHPP polymer when used for prosthetic restoration on implant abutments [5].
The most commonly used materials for the fabrication of conventional prostheses are currently metal alloys titanium. Currently, high-performance polymer biomaterials tend to improve framework properties and potentially reduce the cost of prostheses [6].
In the current study, we investigate the use of polyetheretherketone materials in the prosthetics of different facial fractures in comparison with other materials like titanium.A lot of opportunities and challenges there are in the creation and characterization of biomedical alloys. Features of these materials have been designed to be in contact with blood, as replacements for soft and hard tissues [4].
However, they cannot satisfy the increasing requirements for large-scale production owing to the degradation of metals. Physical surface modification could be an effective way to enhance their bio functionality [2].
Continuous research and development of the polymer industry with applications in all medical fields have their ground in the importance of these biomaterials in the health domain [5].

2. Materials and methods

Polyetheretherketone (PEEK) is a thermoplastic resin employed in the field of industry and medicine for several years. This semi-crystalline high performance composite offers a unique combination of outstanding physical properties, stability at high temperatures and excellent resistance to chemical damage. These are some of the reasons that allow the use of PEEK as a framework material for plates prosthesis, removable dental prosthesis, tooth-implant-supported and implant-supported bridges [7].
A retrospective review of patients hospitalized and treated in the Oral and Maxillo-Facial Surgery Clinic of Craiova had been made.

2.1. Study inclusion criteria

Inclusion criteria were considered to be patients hospitalized and treated in the Oral and MaxilloFacial Surgery department of Craiova hospital for 3 years starting the year 2012. The patient sustained maxillary fractures, either isolated or in combination with other facial injuries. We had a total of 765 subjects. We made 3 groups of distinct patients. The first one was composed of 176 patients with

fractures at both levels of the middle face and the level of the lower face. The other group was made up of 329 patients with lower facial fractures and the last one was represented by 260 patients with middle facial fractures.

2.2. Study exclusion criteria

The most important exclusion criterion had been the time of the study. We used in our study only the patients hospitalized and treated in the Oral and Maxillo-Facial Surgery department of Craiova hospital for a strict period. We also excluded in our study patients with craniofacial malformations, patients with absolute contraindications for surgical procedures such as active malignant tumors, and immuno compromised patients.

3. Results and discussions

For a better evaluation of the cases, we have drawn up a "trauma patient file" in which we had noted data on the etiology of the trauma, the age, gender, the patient's environment, the period from the accident to hospitalization, the topography of the lesion, the type of surgical intervention performed, the method of approach (exo-oral or endo-oral), the evolution, the medicinal treatment administered.

In our research, we grouped patients according to different criteria: age, patients' environment, and gender.The current clinical statistical study set that the frequency of middle-floor fractures between gender is higher in males (73%) than in females (27%). Furthermore, there is an important difference regarding the patients' environment. It had been observed that in the rural environment (58%) the frequency is higher compared to the urban environment (42%). We determined two major ways of producing fractures of the middle level of the face: by aggression or by accident. The greater share had been represented by the fractures caused by aggression (69%) despite the fractures caused by accidents (31%).

An equally important modulating factor of facial fractures is represented by the age of the patients. We set six groups of ages as follows: age between 20-30 years, age between 30-40 years, another group between 40-50 years, 50-60 years, 60-70 years, and the group between 70-80 years. We observed an increased number of fractures in patients aged between 30-40 years (15.23%) and between 20-30 years (13.20%).

Furthermore, the clinical statistical study was carried out based on a batch of 200 patients following the previously described aspects. According to age groups, mandibular fractures predominate in the 20–30-year-old group (58 patients) and occur less often in those over 60 years (13 patients) and between 0-10 years (8 affected patients). The frequency of mandible fractures depending on their cause is dominated by human aggression and road accidents which represent 50%, respectively 37.5% of the total cases. Out of the total of 200 patients with mandible fractures, 170 were male and only 30 were females.

Osteosynthesis plates were chosen for each participant according to the particularities of the fracture. We used titanium plates in a proportion of 95% and polyetheretherketone plates (5%) for the stabilization of the bone fragments. For a 46-year-old patient who presents with comminuted fractures of the right malar bone, a victim of human aggression, osteosynthesis plates were placed, evaluated for the correct location, and fixed to the bone (Figure 1). The surgical approach was mixed (bi-coronary and endo-oral).

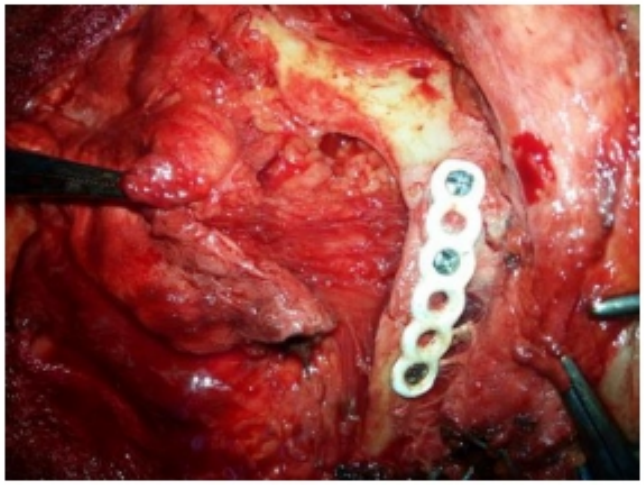


Figure 1. Application of polyetheretherketone plates

Due to the elasticity of the polyetheretherketone osteosynthesis plate, it does not need to be shaped. The plate achieves an effective containment of the fractured fragments (Figure 2).

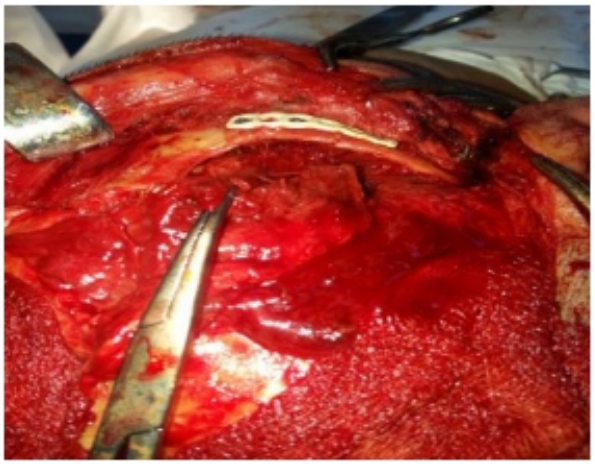
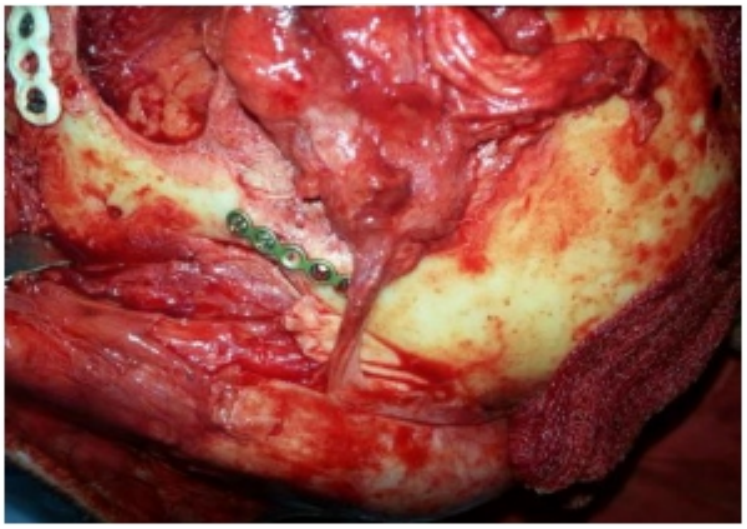


Figure 2. The elasticity of the polyetheretherketone plates

In this case, we decided to use titanium plates and polyetheretherketone plates to compare the results (Figure 3).



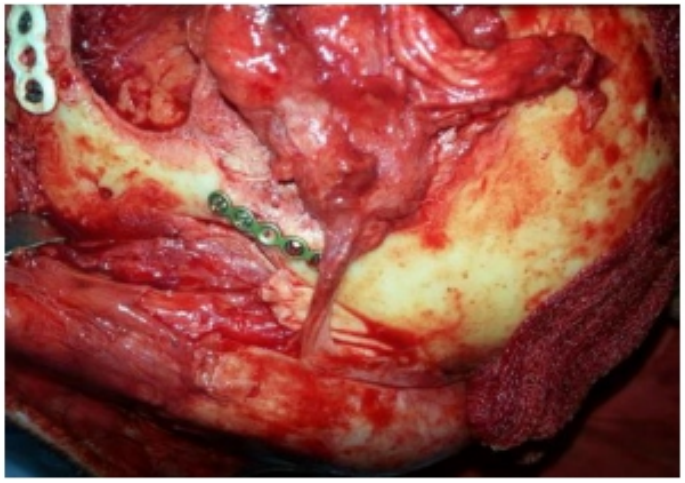


Figure 3. Titanium and polyetheretherketone plates

The locations of the implants were designed to be fitted on the free edges of the fractured bone. The final thickness of the polyetheretherketone plates was reduced after finishing and polishing. The size of the titanium plates was not changed.

Mandibular and maxillary fractures are frequently encountered by the OMF surgeons. Accurate anatomical bone reduction cannot be guaranteed although a lot of techniques and prosthetic devices have been reported for the treatment of fractures. Open reduction internal fixation is the method to obtain almost perfect anatomical reduction, especially in fractures with severely displaced bone segments. In our study we used custom-made polyetheretherketone and titanium miniplates for osteosynthesis to determinate the differences between those materials.

We experienced better results with polyetheretherketone miniplates both because it allows a better customization on the surface of the anatomical defect and because of the better visibility of the fracture edges. It's been beneficially applied as an implant material in various medical fields since the 1990s thanks to its high stability, good biocompatibility, low density, insolubility, excellent fatigue properties, high toughness, corrosion and aging resistance, ease of processing, and color stability [8].

The time spent intraoperatively to fix the bone fracture with the two different types of osteosynthesis plates does not show significant differences, although the polyetheretherketone custom-made implants were by far easier to integrate.

In our study, patients treated with custom-made polyetheretherketone plates did not encounter different local complications than the other group where we used titanium plates. Polyetheretherketone plates were studied for orthopedic surgery, but there are few studies from facial trauma surgery. Thanks to its bone-like elasticity, polyetheretherketone may be a viable different material. Due to its high flexibility polyetheretherketone decreases stresses at the bone–screw interface.

There are studies comparing titanium and PEEK plates that lead to the hypothesis that the elasticity of PEEK fixation may decrease the risk of screw pullout/push [9].

4. Conclusions

Cases treated with the osteosynthesis technique (mini plates and screws) had a favorable evolution, without local complications. Following the surgical intervention, patients were able to resume their daily activities after a short period of hospitalization, without affecting speech, physical appearance, and a normal, non-restrictive diet, thus producing a rapid psycho-social recovery.

Late complications, such as delayed consolidations, pseudarthrosis, mandibular constrictions,

occlusion disorders, or vicious consolidations, were not observed in the treated cases. The advantages of polyetheretherketone plates are the wide possibility of customization depending on the patient, as well as the elasticity comparable to human bone. The titanium alloys used to manufacture the mini plates and screws are biocompatible, easily accepted by the human body, and also compatible with MRI investigations or metal detectors in airports.

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where h is the Planck's constant, ν is the frequency, E0 denotes the single-oscillator energy for electronic transitions and Ed is the dispersion energy.

The results are listed in Table 1, revealing that the addition of the MCNT particles decreases the

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Effect of Polishing Systems on Translucency Parameter of the Resin Composites after Aging

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ABSTRACT

The purpose of this study was to evaluate the effect of finishing/polishing systems on the translucency parameter (TP) of resin composites after accelerated artificial aging (AAA). Four composite resins (Filtek Z250, Admira, IPS Empress Direct, Clearfil Majesty Esthetic) were evaluated. Thirty samples were prepared with each resin composite and divided into three subgroups: control (Mylar strip), disc (Optidisc), and rubber (Dimanto) (n=10). The spectrophotometer was used to determine color measurements. TP was calculated the using the CIEDE 2000 formula. Data were analyzed using Generalized Lineer Model ($p<0.05$). The three factors (composite resin, finishing/polishing, and AAA) had no statistically significant influence on the TP. However, composite and finishing/polishing influenced the TP. Filtek Z250 showed the lowest TP values and the IPS Empress Direct showed the highest TP values before and after AAA. Polished groups showed higher TP values than control groups before and after AAA. IPS Empress Direct with Dimanto had higher TP values than other groups (except for IPS Empress Direct with Optidisc); Filtek Z250 with finishing/polishing groups showed lower TP values than other groups. Composite type and finishing/polishing systems influenced TP values. Unfinished groups exhibited lower TP values than finished groups. There was no difference in TP values between finishing/polishing systems (Dimanto and OptiDisc).

Keywords: accelerated artificial aging, polishing, resin composite, spectrophotometer, translucency parameter

1. Introduction

Due to the recent popularity of esthetic restorative materials and increasing patient demands, color matching of resin composites to natural teeth is necessary for clinicians [1]. The esthetic success of restorations is related to optical properties, such as color and translucency. Restorative materials should have optimal translucency and mimic the natural tooth structure [2]. Translucency can be defined as a property in between opacity and transparency. Translucent materials allow light to pass through them but scatter light compared to transparent materials so that objects on the other side are not clearly visible [3]. Translucent esthetic materials ensure color matching to adjacent tooth/restoration. The translucency parameter (TP) is used to evaluate the translucency of dental materials [4]. The literature recommends the CIEDE 2000 formula for improved correction between perceived color differences obtained from the CIELAB formula [5]. However, recent studies have calculated the TP using the CIELAB formula [4].

The surface layer of the resin in contact with oxygen does not undergo polymerization and requires removal. Application of finishing/polishing systems increases the esthetic appearance and longevity of dental restorations [2]. Different finishing/polishing systems are available for finishing and polishing resin composite restorations. These systems may require one or more steps. Moreover, the abrasive particles differ in composition, presentation, type, and hardness.

Considering that simplified systems save time, clinicians need to be familiar with the materials they use [6]. Finishing/polishing procedures aim to adjust occlusion, create a smooth, uniform surface, and allow adequate light reflection [2]. In clinical terms, restorative materials should be resistant to factors, such as temperature change, exposure to moisture, and mechanical stresses. Clinical studies are required to validate the treatment procedures, but these studies are expensive and time-consuming. In vitro studies may simulate conditions that closely resemble the clinical environment [7]. However, studies involving composites subjected to accelerated artificial aging (AAA) are difficult to compare due to differences in used methods. AAA imitates the effect of prolonged exposure to environmental conditions through an accelerated weathering process that includes differences in light, temperature, and humidity and ensures that AAA in short time intervals is equivalent to long-term use under normal conditions [8].

Although there are concerns on changes in translucency of composite resins after light-curing and finishing/polishing, [9] they have not been adequately researched. To the best of our knowledge, data comparing the effects of multi-step and one-step finishing/polishing systems and AAA on the translucency of resin composites are not available in the literature. Therefore, this study aimed to evaluate the influence of polishing systems on the translucency of resin composites after accelerated artificial aging. The following null hypotheses were investigated: (1) different polishing methods do not influence translucency; (2) the effect of AAA does not influence the translucency of resin composites.

2. Materials and methods

Shade equivalent A2 of four resin composites [(Clearfill Majesty Esthetic, Kuraray, Okayama, Japan; IPS Empress Direct, Ivoclar Vivadent, Schaan, Liechtenstein; Filtek Z250, 3M-ESPE, St. Paul, MN, USA; Admira, Voco, Cuxhaven, Germany)] were used in this study (Table 1).

Table 1. List of materials used in present study

	Manufacturer	Type	Composition		Lot No.
Filtek Z250	3M Espe, St. Paul, MN, USA	Universal/ Microhybrid	BisGMA, UDMA, Bis-EMA, zirkonium/silica, 0,01-3,5 µm	%82 wt %60 vol	NF43622
Admira	VOCO GmbH Cuxhaven, Germany	Ormocer	Ormocer, BisGMA, UDMA, Aromatik ve Alifatik dimetakrilat, 0.7 µm.	%78 wt. (%56 vol. microfiller)	1914502
IPS Empress Direct (Enamel)	IvoclarVivadent, Schaan, Liechtenstein	Nanohybrid	BisGMA, UDMA, TEGDMA, Barium glass, ytterbium trifluoride, and mixed oxides silicon dioxide, copolymer 0,4 µm-100 nm	%75-79 wt %52-59 vol	Y35243
Clearfil Majesty Esthetic	Kuraray Noritake Dental Inc., Okayama, Japan	Nanohybrid	BisGMA, hydrophobic aromatic dimethacrylate, di-Camhorquinone, silanated barium glass filler, pre-polymerized organic filler, 0,37 µm-1,5 µm	%78 wt %40 vol	4H0173
Finishing/Polishing systems					
	Manufacturer	**Type**	**Composition**		**Lot No.**
OptiDisc	KerrHawe, Bioggio, Switzerland	Discs	Aluminum impregnated discs, (Coarse-Medium-Fine-Extrafine)		6778506
Dimanto	VOCO GmbH Cuxhaven, Germany	Rubber	Diaomond particles impregnated silicon rubber (One-step pre and high gloss p olishing)		1915625

Thirty disc-shaped samples were prepared for each composite (total 120 samples). A Teflon mold (8 mm diameter, 2 mm thickness) was used to prepare disc-shaped specimens of resin composites. Resin composites were placed into a hole, and a Mylar strip was placed over the top surface.

Resin composites were cured using LED (3M Elipar™ Deep Cure- S LED, Saint Paul, MN, USA) for 40 s directly over the Mylar strip. Resin composite groups were randomly divided into three subgroups (n=10). Except for the Mylar strip groups, 1200 grit silicon carbide abrasive paper was used with water before application, using the polishing systems. Two polishing systems [(Optidisc, KerrHawe, Bioggio, Switzerland; Dimanto, Voco, Cuxhaven, Germany)] used are shown in Table 1. The four-step OptiDisc system includes four aluminum oxide (Al2O3)-embedded discs, each used for 15 s, under dry conditions. The one-step Dimanto system (rubber cup), with diamond-embedded rubber cups, was used for 60s underdry conditions. The polishing systems were applied using a handpiece at a speed of 10,000 rpm. All specimens were rinsed for 10 s and stored at 37°C for 24 h in distilled water.

2.1. Translucency parameter

The initial color measurements were obtained using a spectrophotometer (Lovibond RT Series, Tintometer® Group, Lovibond House, UK) calibrated according to the manufacturer's instructions. TP was calculated using the CIEDE 2000 formula [4]:

$$TP_{00} = \left[\left(\frac{L'_B - L'_W}{K_L S_L}\right)^2 + \left(\frac{C'_B - C'_W}{K_C S_C}\right)^2 + \left(\frac{H'_B - H'_W}{K_H S_H}\right)^2 + R_T\left(\frac{C'_B - C'_W}{K_C S_C}\right)\left(\frac{H'_B - H'_W}{K_H S_H}\right)\right]^{1/2}$$

Subscripts "B" and "W" (specified in the formula) correspond to black and white backgrounds, respectively. (LB′- LW′), (CB′- CW′), and (HB′- HW′) denote the differences in lightness, chroma, and hue on black and white backgrounds, respectively. The relationship between the variations of chroma and hue in the blue region is defined by the rotation function (RT). The weighting functions of lightness, chroma, and hue are denoted by SL, SC, and SH, respectively. KL, KC, and KH consist of parametric factors set 1 in this study [4].

2.2. AAA procedure

After initial measurements, all specimens were aged for 300 h and 150 kJ/m2 in an accelerated aging chamber (Atlas ci 4000; Atlas Electronic Devices Co., Mount Prospect, Il, USA) The aging procedure was performed as stated in the previous study [8]. The aging process was as follows: 60 min in the dark with back water spray; 40 min under illumination; 20 min under illumination water spray; and 60 minunder illumination. The temperature of the back panel was maintained at 38 ± 2°C in the dark and 70 ± 3°C under illumination. The dry-bulb temperature was 38 ± 2°C in the dark and 47 ± 3°C under illumination. Relative humidity was maintained at 95 ± 5 % in the dark and 50 ± 5 % under illumination. After the AAA procedure, the procedures for measuring TP were repeated.

2.3. Statistical analysis

Statistical analysis was performed using the IBM SPSS Statistics for Windows (Version 23.0. Armonk, NY: USA) package program. The data were checked for normal distribution (Kolmogorov Smirnov test for skewness and kurtosis). General linear model was used for TP of interaction between the factors (group × composite × AAA). Bonferroni correction was used to compare the main effects.

Tukey's test was used for multiple comparisons. The significance level of difference was set at 0.05.

3. Results and discussions

Interaction factors are shown in Table 2 for TP. The analyzed factors (resin composite, finishing/polishing, and AAA) had no statistically significant influence on TP. However, resin composite and finishing/polishing systems influenced TP. TP values are shown in Table 3. The lowest TP values were found with Filtek Z250 and the highest TP values with IPS Empress Direct before and after AAA.

Compared to polishing systems, the lowest TP values were found in control groups before and after AAA. There were no differences within Optidisc and Dimanto groups before and after AAA. IPS Empress Direct with Dimanto had higher TP values than other groups (except IPS Empress Direct with Optidisc). Filtek Z250 exhibited lower TP values than other resin composites.

Table 2. Interactions among the factors for TP

	Type III		
	Wald Chi-Square	**df**	**Sig.**
(Intercept)	23042.985	1	<0.001
Group	37.359	2	<0.001
Composite	333.908	3	<0.001
AAA	3.762	1	0.052
Group * Composite	33.882	6	<0.001
Group *AAA	0.022	2	0.989
Composite * AAA	0.648	3	0.885
Group * Composite *AAA	0.079	6	1

Table 3. Means and standard deviations for TP values

		Composites				
Groups	Time	Filtek Z250	IPS Empress	Admira	Clearfil M	Total
Control	t1	5.32 ± 0.48	6.58 ± 0.94	6.32 ± 0.90	7.18 ± 0.62	6.35±1.00
	t2	5.17 ± 1.29	6.45 ± 0.86	6.25 ± 0.51	6.90 ± 0.24	6.20±1.02
	Total	5.25 ± 0.95^{a}	6.52 ± 0.88^{bef}	6.28 ± 0.71^{e}	7.04 ± 0.48^{cb}	6.27 ± 1.00^{A}
Optidisc	t1	5.37 ± 0.53	7.70 ± 0.64	6.94 ± 0.58	7.21±0.45	6.81±1.03
	t2	5.22 ± 0.63	7.57 ± 0.75	6.81 ± 0.37	6.96±0.45	6.64±1.03
	Total	5.30 ± 0.57^{a}	7.64 ± 0.68^{cd}	6.87 ± 0.47^{bef}	7.08 ± 0.46^{cb}	6.72 ± 1.03^{B}
Dimanto	t1	5.48 ± 1.09	8.16 ± 0.59	7.14 ± 0.73	7.24 ± 0.69	7.00±1.24
	t2	5.29 ± 0.78	7.95 ± 0.59	7.09 ± 0.76	6.94 ± 0.68	6.82±1.19
	Total	5.38 ± 0.93^{a}	8.05 ± 0.59^{d}	7.11 ± 0.73^{ef}	7.09 ± 0.68^{cb}	6.91 ± 1.21^{B}
Total	t1	5.39 ± 0.73	7.48 ± 0.98	6.80 ± 0.80	7.21 ± 0.57	6.72 ± 1.12^{X}
	t2	5.23 ± 0.91	7.33 ± 0.96	6.72 ± 0.65	6.93 ± 0.47	6.55 ± 1.10^{X}
	Total	5.31 ± 0.82^{A}	7.40 ± 0.97^{B}	6.76 ± 0.73^{C}	7.07 ± 0.54^{D}	6.64±1.11

The current study found significant differences between polishing systems on TP. Therefore, the ull (first) hypothesis was rejected. AAA did not influence TP. Therefore, the null (second) hypothesis was accepted. In the current study, resin composites were not colored to any agents. Translucency is influenced by various factors; the thickness of composites, [10] pigments and other chemical ingredients of the material [11] and light-curing protocol [12]. This study used composite samples of A2 tons and equal thickness. The same light-curing device was used for the polymerization of samples. Due to theshortcomings of the CIELAB formula in the literature, we used the CIEDE 2000 formula [4] to calculate TP values. The optical characteristics of teeth depend on the region, between the teeth, and factors, such as the choice of material. Ideally, the color and translucency of restorative materials should mimic natural teeth in esthetic restorations [8].

The ability of a material to allow passage of light is an indicator of translucency, and higher TP values show higher translucency [2]. Salas et al. [4] reported the translucency perceptibility and acceptability thresholds of composite resins according to the CIEDE 2000 formula as 50%:50% TPT (perceptibility) 0.62 and TAT (acceptability) 2.62, respectively. Our study results showed that changes in TP values of the materials were clinically acceptable. TP values of resin composites decreased after AAA. However, this decrease was not significant, probably due toincreased reflectance of higher ΔL values [2]. TP differences in samples can be attributed to compositeresin contents and the effect of polishing systems. Lee and Lee [13] reported that the translucency of resin composites increased after polishing. In the current study, control groups showed lower TP values than finished/polished groups. These findings contradict the results of a study byElsayad [2]. A previous study showed that the multi-step system increased TP values; however, the one step system decreased TP values, with significant TP change [14]. Our study showed no difference in TP values between finishing/polishing systems. Different application and aging protocols used in studies may affect results. Moreover, applied polishing systems can be crucial to material translucency and scattering from the surface because they increase surface gloss, resulting in higher TP values than control groups.

A previos study show that nanohybrid composite resins have high translucency due to particle sizes smaller than the wavelength of light, resulting in minimal scattering of photons [15]. In current study, IPS Empress Direct groups showed higher TP values than other groups, probably due to the differentiation of light scattering resulting from nano-sized filler particle of the IPS Empress Direct composite is used in the anterior region. In this context, its translucency is expected to be high. CeramX Duo and Admira are ormocer-based composite resins. Cengiz et al. [16] stated that TP values were low due to ormocer content. However, our study showed the lowest TP values for Filtek Z250. Optical properties of resin composites are affected by resin matrix composition, pigment, and other addedsubstances, which result in light reflection at different wavelengths [17]. Naeimi Akbar et al.[18] stated that translucency values of resins increase with a decrease in filler particle size and volume. In our study, Filtek Z250 had higher filler volume and particle size than other materials that may have played a role in low TP values. Howard et al. [19] reported that the differences in refractive index between filler and matrix decrease with an increase in the C=C conversion degree of monomers so that the resin scatters more light and shows more translucency. Material composition differences can be attributed to variations in TP values. Reports show that the translucency values of the materials are affected by many factors, [4] such as the composition of the applied resin matrix, [4] resin matrix content, [20] distribution of filler, [21] number of particles per volume, different chemical structures, added substances,[8] and types of polymerization initiator/inhibitor [21]. In resin composites, light absorption is enabled by the organic matrix, while diffusion is due to the size and distribution of inorganic fillers and the difference between the refractive index of the organic matrix and inorganic filler contens[14, 22]. Azzopardi et al.[20] stated that resin matrix and filler particles could influence on the translucency of experimental compositeresins. Furthermore, the translucency of resins depends on absorption and scattering, although scatteringoccurs due to the refractive index mismatch between the organic matrix and filler particle and the size and dispersion of inorganic filler [22].

This study subjected composite resin samples to polishing systems after 300 h AAA and found differences in TP values. Within the methodological limitations, to mimic the effects of aging on materials that may occur in the oral environment in a short time to estimate the clinical performance of resin composites. The oral environment can influence the longevity on translucency in resin composites.

Further studies using different aging or finishing/polishing methods are necessary to complement the currently obtained results. This study may contribute to improved techniques for preserving the translucency of resin composites.

4. Conclusions

AAA did not influence TP values. Composite and finishing/polishing systems influenced TP values. Unfinished (control) groups exhibited lower TP values than finished groups. There was no difference in TP values between multi-step and one-step groups. The highest TP values were noted with IPS Empress Direct (nanohybrid) and the lowest TP values with Fitek Z250 (microhybrid) before and after AAA.

Clinicians should be aware that the polishing systems they use may affect the translucency values of resin composites.

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Composites Based on Sustainable Biomass Fiber for Automotive Brake Pads

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ABSTRACT

Biomass fibers are promising materials for applications in modern vehicles. They have great economic and ecological significance, as well as a great potential in the fabrication of composite materials due to the relatively high level of strength and rigidity, low density, availability, recyclability, and biodegradability. In this context, the focus is on the development of automotive brake pad materials from sustainable sources. This work refers to the investigation of the behavior of composite materials made of biomass fibers, phenolic resin, graphite and aluminum oxide. These materials are intended to be used for brake pads on automobiles with moderate efficiency. For this purpose, three recipes of composite materials with different percentages of coconut fiber and wood powder were developed in laboratory. The physical and mechanical as well as functional properties of these composite materials with varying amounts of biomass fibers are examined in this paper. The best performances in this terms was obtained for the composite material containing the highest amount of wood powder and the lowest amount of coconut fiber.

Keywords: fiber, brake pad, biomass, coconut, wood powder

1.INTRODUCTION

In the braking process of motor vehicles, brake pads play a particularly role in speed control. Their appearance was a welcome idea in the industry, but the dust, fine and ultrafine particles that they release outside following the braking process represent a great concern currently [1]. Over time, the construction of brake pads has evolved both in terms of the materials and in terms of the impact on the [2]. Asbestos-based brake pads are no longer allowed for use in vehicles because of their carcinogenic properties [3]. In this context, environmentally, friendly and non-toxic materials are gaining popularity among researchers in all industries. Introduction of environmentally and friendly natural fibers to replace asbestos in the production of brake pads has become a popular concept among researchers in the automotive industry. The technologies of the future are based on the principles of sustainable design, which means the use of energies from renewable sources, the rational use of natural resources, the development of new materials with superior properties, capable to elimination of waste.

The valorization of resources from waste is a current concern of society, so the creation of new materials

The valorization of resources from waste is a current concern of society, so the creation of new materials and technologies based on waste as raw materials, especially vegetable ones, is a field that requires immediate attention [2]. Fibers derived from agricultural waste have great economic and ecological significance, as well as a great potential in making composite materials due to the relatively high level of strength and rigidity, low density, availability, durability, recyclability, biodegradability [4]. In this context, the production of brake pads from sustainable sources is required [5]. European Union strategies try to reproduce the specific objectives and means by which waste management methods can be improved for a better use of natural resources [6]. Composite materials based on biomass fibers are the ones we must focus on in the future, not only because they have proven their reliability over time, but especially because they come from renewable sources. These materials through their properties can successfully replace expensive materials from non-renewable resources. In the specialized literature there are several studies that analyzed different combinations of materials for brake pads asbestos free.

Thus, many researchers have succeeded in improving the performance of braking systems by introducing composite materials produced from sustainable fibers.
Idris et al. produced brake pads made with banana peel waste and studied the influence of phenolic resin content on their braking performance, [7]. Daut et al. studied brake pads made from coconut powder [8].
The behavior of composites for brake pads using different combinations of biomass fibers has also been analyzed in the specialized literature.Juan et al. produced brake pads with different compositions of coconut shell, walnut shell, pineapple leaf, carbon and polyurethane resin using powder metallurgy. Experiments shown that the produced friction material has an improved braking performance, and the polluting emissions resulting from braking are low, [9]. Rajmohan et all. tested natural sugar cane fibers and coconut fiber in the manufacture of brake pads. These were combined with epoxy resin, SiC powder. Experiments showed an improvement in tribological parameters with increasing coconut shell content, [10]. Sutikno et al.produced friction material with bamboo fiber, coconut, alumina, magnesium oxide and epoxy resin. The braking performance achieved was superior to commercial brake pads, [11]. Another combination of fibers from agricultural waste used in the production of brake pads was analyzed by Kholil et all. They produced brake pads from natural coconut fiber and waste wood powder used in the braking system of motorcycles, [12].

In this paper is analyzed the behavior of composite materials made of biomass fibers, phenolic resin, graphite and aluminum oxide intended for brake pads used for small vehicles with medium performance, which are harmless to human health. For this purpose, three recipes of composite materials with different percentages of coconut fiber and wood powder were developed. The paper analyzes the physical mechanical and functional characteristics for these recipes with different proportions of biomass fibes.

2. Materials and methods

Biomass from agricultural production has commercial and environmental value and can be put to use in the fabrication of brake pads, [2]. It can be used as reinforcement in composite materials. In order to obtain the friction material, several materials must be chosen which are mixed with a resin with the role of binder with the property of transforming thermoplastic into thermoset.In this paper, will be produced and characterized friction materials for brake pads used for small vehicles with medium performance, which are composed of coconut fiber and wood powder.

Coconut fiber comes from the shell of the exotic fruit, and wood dust is a waste that comes from factories that use wood in the production process. In order to obtain efficient composite materials with predictable properties, an important role is played by the chemical composition of the fibers in order to achieve strong connections between the fiber and the resin. Table 1 shows the chemical composition of coconut fibers, and Table 2 shows chemical composition of wood powder.

Tabel 1. Chemical composition of coconut fibers

Cellulose (%)	Hemicellulose (%)	Lignin (%)	Pectin (%)	Water (%)
33.07	8.5	39.23	8.12	11.08

Tabel 2. Chemical composition of wood powder

Cellulose (%)	Hemicellulose (%)	Lignin (%)	Pentosan (%)	Ash (%)	Water (%)
40.81	29.12	20.23	5.43	1.01	3.4

By comparing Tables 1 and 2, we can deduce that the main components of biomass fibers are cellulose, hemicellulose, lignin, protein, pectin, and ash. Glycosidic bonds in cellulose play a decisive role in determining the composite's mechanical characteristics. Hemicellulose is a component of the plant cell wall and is made up of several monomers such as: xylose, galactose, mannose, rhamnose and arabinose that lead to the formation of a branched and amorphous polymer. Lignin is a phenolic compound that imparts compressive strength and stiffness to the plant body.

Humidity is a characteristic of vegetable fibers, and its content must be determined precisely, for each type of fiber. An excess of humidity can lead to damage to the material that contains it. The literature shows that for various vegetable fibers the water content varies between 9 and 12%, [13].

Regarding the water content of the wood powder that will be used in the recipe, it is less than 4%.

In order to produce the composite materials in the laboratory, the biomass fibers were chemically treated with a 5% alkaline solution. The role of this surface treatment is to achieve optimal bonding interfaces between the fiber and the organic matrix, by removing deposition materials and activating the fiber surface.

The following materials were used in the development of composite materials: coconut fiber, wood powder, phenolic resin, graphite, aluminum oxide. The coconut fibers and wood powder are used as reinforced material. The binder used in the production of composite materials was phenol formaldehyde resin. Graphite used in this study as friction modifier. The aluminum oxide is used as abrasive. Figure 1 illustrates different forms of the materials that are used in development of a composite materials. An increased level of homogeneity in the recipe's ingredients is essential for producing products with better physical-mechanical and functional. An increased level of homogeneity in the recipe's ingredients is essential for producing products with better physical-mechanical and functional attributes [14-20].

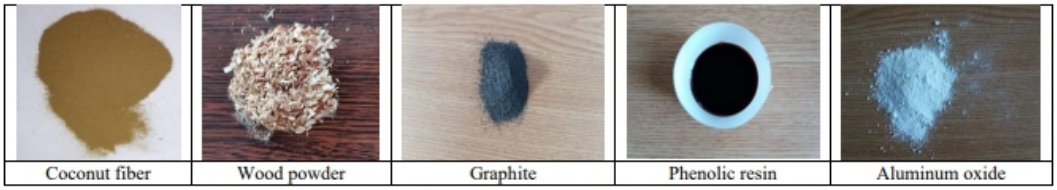

Figure 1. Raw materials used to obtained composite materials

With the raw materials shown in Figure 1, were developed three material recipes with different concentration of coconut fibre and wood powder (Table 3).

Table 3. Chemical composition of composite materials

Samples	Coconut fiber (%)	Wood powder (%)	Graphite (%)	Phenolic resin (%)	Aluminum oxide (%)
A	20	40	5	25	10
B	30	30	5	25	10
C	40	20	5	25	10

The technological flow for the laboratory production of composite materials with biomass fibers includes the following stages: procurement of fibers, grinding, drying, chemical surface treatment with alkaline solutions, resin application, mixing, hot pressing, sintering, product finishing.
The biomass fibers together with the other materials in the recipes were mechanically mixed for 10 min, after which the formaldehyde resin was added together with a small amount of sulfuric acid as a catalyst. In order to homogenize the components of the recipes, they were mixed using a mixer with a power of 500W and a speed of 2800 rpm. This mixture was placed in a circular mold with a diameter of 96 mm in which the mixture was pressed (Figure 2).

Figure 2. Mold with composite material during the pressing process

Figure 3. Disc samples made from developed recipes

Table 4 shows composite fabrications details. Figure 3 shows the samples obtained at the end of the manufacturing process.

Table 4. Composites fabrications details

Sintering conditions	Temperature (°C)	Pressure (MPa)	Time (min)
Conditions for molding	150	15	10
Conditions for oven curing	200	-	300

The samples obtained after the three recipes were adequate in terms of compactness, integrity, elasticity and appearance when removed from the mold. For the three recipes of composite materials, were determined the physical-mechanical and the functional characteristics.

3. Results and discussions

3.1. Physical and mechanical characteristics

Determination of the density of the new materials was used fluid displacement method. The samples were immersed in a cylinder with distilled water. The mass of the samples, was determined by weighing with a balance that has an accuracy of ±0.01, before and after their immersion in water. The volume of the piece was determined by the difference between the volume of water before and after immersion.For each sample were made three readings, taking into account the average value. The density of the samples was calculated, the results being presented in Table 5.

Water absorption was determined for the three proposed recipes. The samples were measured, then immersed in water for 24 h at ambient temperature before being weighed again. Moisture was removed and samples were reweighed after this period. The difference in mass gave the amount of water absorbed and their percentage.

Vickers hardness according to ASTM 92 was determined for each sample. The average results from three separate trials of each samples were used to determine the final valuation (Table 5).

The compressive strength was determined according to SR EN 1926:200795 and SR EN 1926:200796 standards. The measurements were carried out with a universal machine for the static testing of materials. The results are determined as average values of three separate measurements (Table 5).

Table 5. Physical and mechanical characteristics of composite materials

Samples	Density (g/cm^3)	Water absorption (%)	Hardness VHN	Compression strength (MPa)
A	2.55	5.22	56	176.7
B	2.78	4.99	49	140.3
C	2.95	3.76	45	162.6

The lowest density was obtained for sample A. This is explained by the fact that a porous material was obtained as a result of the combustion of cellulose during the manufacturing process, which reduced the density of the finished product.

The literature also states that in these conditions the braking noise is also reduced, which is an important thing in the operation of the brake pads [3]. As a result of the increased porosity of sample A, it also had the highest percentage of water absorption. On the other hand, sample A has the highest concentration of wood fiber that contains the highest amount of cellulose, which is why it absorbs water better. The creation of hydrogen bonds among hydroxyl groups and water explains how lignocellulosic materials are able to absorb water, [14]. Regarding the hardness of the composite materials, an increase is observed as the concentration of wood powder increases. Sample A has a 56VHN hardness, which is the highest of the samples tested. Hardness decreases with increasing coconut fiber composition and reducing wood dust. Consequently, samples B and C with a higher amount of coconut fibers have a

lower hardness. The lowest compressive strength was sample C in which there is an equal amount of coconut fiber and wood powder that resulted in a uniform matrix. The highest compressive strength was recorded for sample A.
The physical and mechanical properties determined experimentally for the types of materials presented are influenced by several parameters such as: the proportions of raw materials used, the nature of vegetable fibers, the fiber-matrix interface, alkaline treatments applied, the parameters of the manufacturing technology. In order to choose the composite material with superior characteristics, it is also necessary to evaluate the functional characteristics.

3.2. Tribological characteristics

The coefficient of friction, few wear parameters, and temperature at the friction couplings were all tracked over time to evaluate the tribological performance of the laboratory-made composite materials.
Dry friction conditions were used using a TR-20 tribometer, whose working principle is focused on the "pin on disc" approach, to calculate the friction coefficient over time. The equipment's pin is a steel ball 6 mm in diameter, and each of the three composite material samples is 30 mm by 30 mm by 20 mm.
Experiments used a pressing force of 10 N. Table 6 lists the various experimental conditions.

Table 6. The parameters for determining the friction coefficient

The diameter of the wear mark (nm)	Speed (rot/min)	Test time (h)	Test distance (m)
5	150	5	2200

The friction coefficient of the samples has a relatively slow increase over time, and its value stabilizes until the end of the test period.
According to specialized literature, Braking requires a friction coefficient that is consistently greater than normal, hence friction materials must be designed accordingly [15, 16]. The evolution of the friction coefficients for the composite materials tested is presented in Figure 4.
Shortly after the beginning of the trials, in the vicinity of 2000s, the friction coefficient of sample A was reached 0.4. Sample B's friction coefficient reached a maximum of 0.35 after 14,000 s, and its minimum was 0.3 after 18,000 s. A higher percentage of wood powder and a lower percentage of coconut fiber resulted in a higher friction coefficient, as seen in Figure 4.

To determine the wear of the composite materials, they were tested under dry friction conditions, using the pressing force F=10N and two sliding speeds: 3.92 m/s and 4.71 m/s. The test time was 10 min.
The wear of the investigated materials is evaluated using the gravimetric technique. [18, 19].

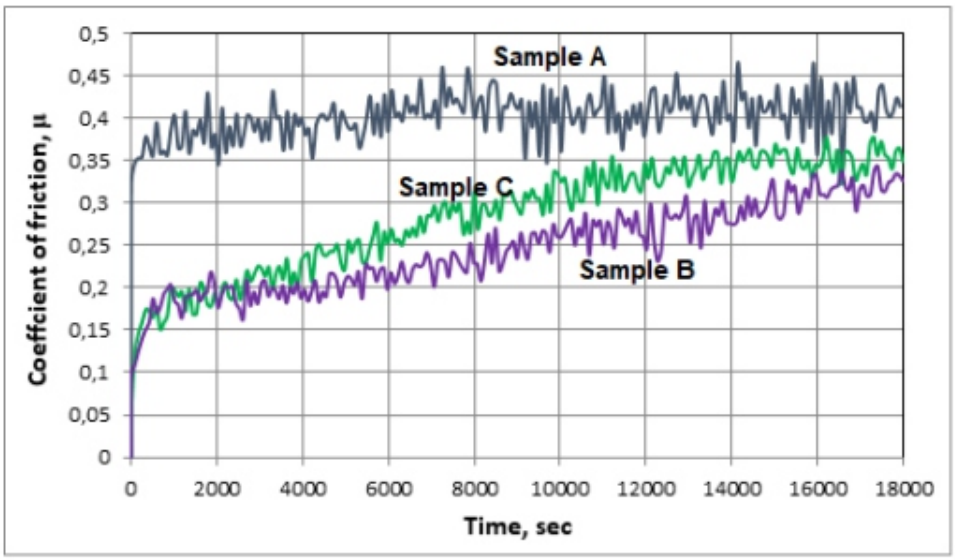

Figure 4. Evolution of friction coefficients for samples A, B and C

In this sense, the mass wear and the linear wear rate are determined, the results being presented in Table 8. The wear rate was determined using the formula: [18-20]:

$$W_e = \frac{\Delta_m}{F \cdot L} \qquad (1)$$

In relation (1) m - the difference between sample mass at the beginning and sample mass at the end [g]; F - the normal force applied to the tribometer, [N]; L - sliding distance, [m].

Table 8. Samples wear rate

Samples	Sliding speed v [m/s]	Initial mass m_i, [g]	Final mass m_f, [g]	Mass wear Δ_m, [g]	Wear rate [g/N·m]
A	3.92	46.3162	46.306	0.0102	$4.63 \cdot 10^{-7}$
	4.71	45.6965	45.6882	0.0083	$3.77 \cdot 10^{-7}$
B	3.92	52.2135	52.1848	0.0287	$13.4 \cdot 10^{-7}$
	4.71	53.0911	53.0667	0.0244	$11.09 \cdot 10^{-7}$
C	3.92	54.3310	54.3178	0.0132	$6.0 \cdot 10^{-7}$
	4.71	55.5260	55.5168	0.0092	$4.18 \cdot 10^{-7}$

As seen in Table 8, increasing sliding speed reduces the linear wear rate. The wear rate was lowest in Sample A, which had the most wood powder. The rate of wear was greatest in Sample B, when the proportions of coconut fiber and wood powder were balanced. This is because the optimal bonding qualities, in addition to the ratio of materials, determine the best wear rate. The composites' high wear resistance comes from the natural fibers' strong bond to the resin.

As seen in Table 8, increasing sliding speed reduces the linear wear rate. The wear rate was lowest in Sample A, On the other hand, the higher wear resistance of sample A is due to the high cellulose content of the wood powder. This can only be achieved through careful formulations of selected materials in the right proportions.

In order to test the organic material produced in the laboratory, friction couplings' contact regions had their heat fields examined on an experimental installation that allows testing under intensive braking.

To carry out the experiments, brake pads were made from each recipe developed and were mounted in the installation. Figure 5 shows the brake pads made according to recipes A, B and C, and Figure 6

shows the experimental installation. Experiments are based on which the temperature is recorded with a thermographic camera after each of 10 separate braking events. According to specialized literature, the temperature after the ten successive braking must be lower than, $\tau_s \leq 300^0 C$ 300 , [20]. The brake disc in the installation is a ventilated one made of cast iron, brand G2500 according to ASTM A 159 norms intended especially for road vehicles.

A thermographic camera was used to measure the temperature of the brake pads where they made contact with the disc brake.

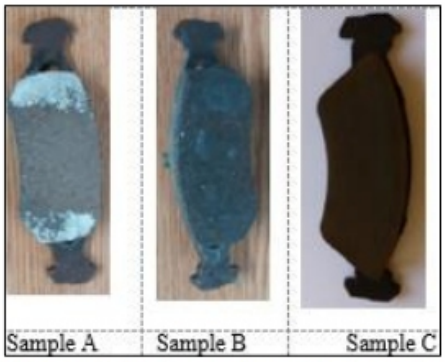

Figure 5. Brake pads made from the composite materials

Figure 6. Experimental installation for intensive braking

Table 9 shows the brake pad temperatures recorded at each braking for the composite materials tested. The initial temperature was 22oC, and successive braking were performed at an interval of 2 s.

Table 9. Temperature values during successive braking

Samples	Pedal power (N)	Temperature during braking τ (^{0}C)									
		1	2	3	4	5	6	7	8	9	10
A	120	24	28.4	36.6	62.7	97.8	133.6	149	245.1	235.3	206.5
B		30	42.1	68.9	89.5	103.8	145.7	178.2	257.9	268.1	269
C		28	37.9	42.3	65.7	99.1	145.7	167.9	256.2	254.9	253.7

For all materials tested, the temperature of the brake pads rises rapidly at the beginning of the test period. This is explained by the fact that part of the heat generated during braking accumulates in the brake pads. After eighth braking, for samples A and C the temperature starts to decrease, this being explained by the fact that it dissipates in the external environment. Sample A had a lower temperature than Sample C after 10 cycles of severe braking. The deterioration of organic components in composite materials is reduced if the temperature throughout the contact region is low.

Also, the temperature during the experiments does not exceed 300oC, which complies with the recommendations in the specialized literature, [19]. The highest temperature accumulated after the ten braking was in sample B, and sample A had the best behavior.Like any research developed on a given theme, it cannot be said that it is completed at this moment, because opportunities for development and deepening can constantly open up, with the resumption of the stages completed in the research under a different approach or by imposing new work techniques.

4. Conclusions

At the end of the previously presented research, Consequently, we may infer the following:
- the use of agricultural waste in the manufacture of friction material intended for making brake pads is a sustainable source of raw materials;
-because biomass fibers are composed of many diverse components, each of which has its own unique qualities, it affect their performance in use:
-because of their high structural quality, the friction materials that are manufactured have excellent physical mechanical properties;
-the composite materials developed in laboratory have a low density compared to the metals;hhardness and compressive strength increase with the increase in the amount of wood powder;
-samples with equal proportions of wood powder and coconut fiber did not give good results in terms of physical-mechanical and tribological characteristics;
-increases in wood powder content and decreases in coconut fiber content lead to a higher friction coefficient;
-sample A, with the best tribological behavior, has the friction coefficient with a value of 0.4;
-the linear wear rate decreases with increasing sliding speed;
-at the end of the ten intensive braking, the temperature in sample A is lower than in sample C, which means that the composite material produced according to recipe A has better ability to dissipate heat into the atmosphere;
-the biomass fibers used had a suitable behavior and can be used as a filler material for the production of brake pads for small vehicles with medium performance, if their proportion in the recipe is adequate.

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The Effect of Disinfection Methods on C. Albicans in Three Types of Denture Base Materials

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A B S T R A C T

This study aimed to evaluate the efficacy of disinfectant solutions on Candida albicans ©. albicans) for different types of denture materials. A total of 144 specimens (10x10x2 mm) were obtained from three different materials: autopolymerized acrylic resin, heat-cured acrylic resin, and hard relining material (n=6) Three disinfectant solutions were used: 100% white vinegar, 2% chlorhexidine digluconate (Saver) and denture cleaning tablets (Corega). The specimens were placed on Eliza plates and 1.5 mL of Yeast Extract Peptone (YPD) was added to each well. Then, 30µL of candida culture was added to the wells. Next, the specimens were incubated at 37°C at 80 rpm for 48 h. Disinfectants were added to the Eliza plates. For all specimens, the disinfectants were replaced with 2 mL of sterile water and kept at 100 rpm for 30 min. Then, 0.1 mL of the liquid was taken and inoculated into the pads containing YPD medium. After incubation, the candida colony growth on the pads was measured. Scanning electron microscope (SEM) images were taken from randomly selected specimens from each group. Statistically significant differences (P<0.05) were found between the disinfectant method groups and the control group for the three types of denture materials. The 2% chlorhexidine gluconate (Saver) disinfectant was the most effective for C. Albicans ATCC 60193 and oral isolate of C. Albicans for all three of the tested denture materials. The effect of cleaning of tabs (Corega) and white vinegar was found to be similar.

Keywords: Candida albicans, vinegar, chlorhexidine digluconate, denture cleaning tabs

1.Introduction

Denture stomatitis is one of the most common types of oral fungal infection [1]. The most common causes of stomatitis are poor oral hygiene and poorly fitting removable dentures [2]. The etiology of denture stomatitis includes local and systemic factors [3]. Despite the multifactorial etiology, the main cause is Candida spp., especially Candida albicans (C. albicans) [4]. C. albicans is a candida species in the human oral microflora [5].

Denture stomatitis is difficult to treat [6]. To reduce the risk of this disease, oral and denture hygiene should be improved [6], antiseptic or disinfectant solutions should be applied to dentures regularly [3,6-9], and dentures should be relined or replaced [3,8]. Moreover, treatments may be required, which include topical and/or systemic antifungal drugs [3,8].If patients who use dentures cannot practice adequate oral hygiene, it is difficult to control the infection. Therefore, disinfectant solutions reduce to need to use systemic antifungal drugs, which has been recommended to prevent denture stomatitis and

to protect oral microbiota [10-12].The present study aimed to compare three types of disinfectant solutions: easily available 100% white vinegar, 2% chlorhexidine digluconate solution (Saver), and denture cleaning tablets (Corega).

The null hypothesis of this study was that the 2% chlorhexidine digluconate (Saver) solution is the most effective treatment for all types of denture materials.

2.Materials and methods

A total of 144 specimens were obtained from three different materials: autopolymerized acrylic resin (S.C. Self Cure, Imicryl, Konya, Turkey), heat-cured acrylic resin (I.Q.-15 Heat Cure, Imicryl, Konya, Turkey), and hard relining material (Ufi Gel Hard, Voco, Cuxhaven, Germany) (n = 6). Three disinfectant solutions were used in the study: 100% white vinegar (Taris Tarpak, İzmir, Turkey), 2% chlorhexidine digluconate (Saver), and denture cleaning tablets (Corega).

The dimensions of the prepared wax specimens were 10x10x2 mm. All of the wax specimens for the heat-cured acrylic resin were placed in the muffle furnance and negative spaces were obtained. Then, the monomer and liquid were mixed in a glass container in accordance with the manufacturer's instructions. Finally, the heat-cured acrylic specimens were prepared by placing them in the muffle furnance.

Silicone molds were used for the autopolymerized acrylic resin and the hard relining material, and specimens, with dimensions of 10x10x2 mm, were prepared using liquid/powder in accordance with the manufacturer's instructions.

One specimen from each group was selected and scanning electron microscopy (SEM) images were obtained before the three disinfectant methods were applied (Figure 1).

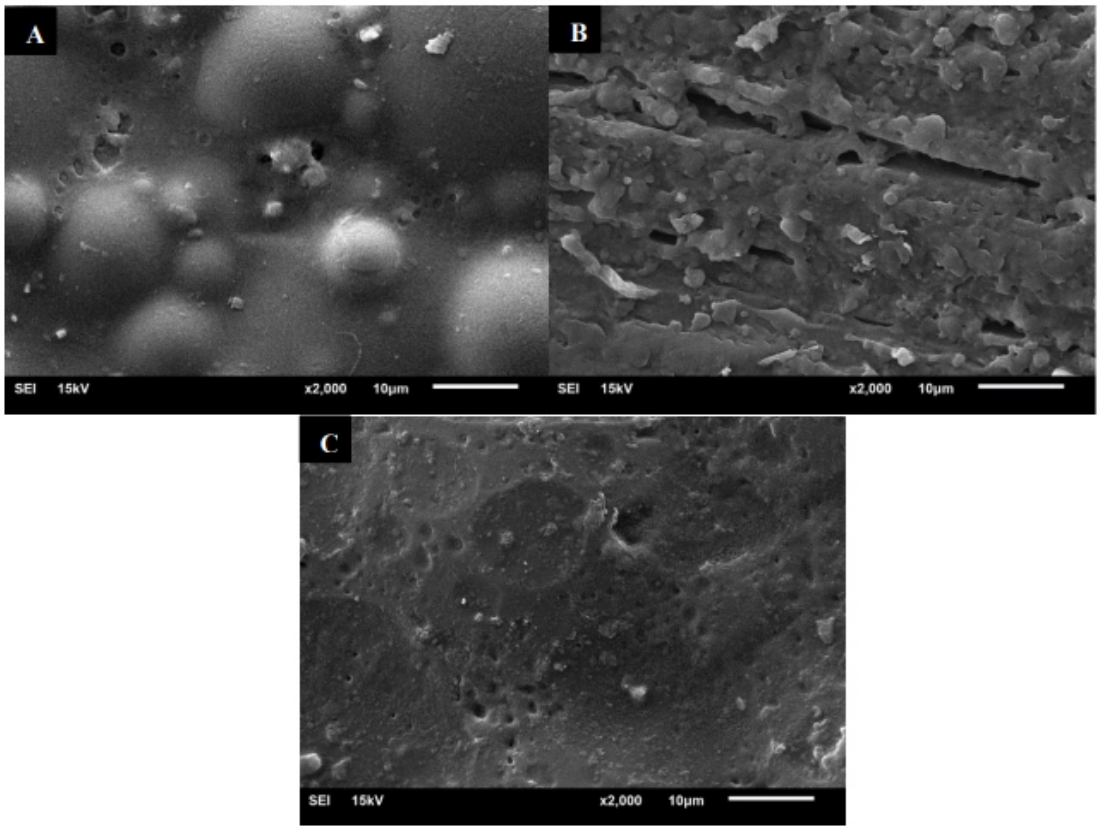


Figure 1. SEM images before the C. albicans strains were applied. (A) autopolymerized acrylic resin, (B) heat-cured acrylic resin, (C) hard relining denture material

Next, C. albicans ATCC 60193 and an oral isolate of C. albicans were reactivated. A suspension of Mc. Farland 0.5 / x107-8 cfu/mL C. albicans was prepared. The optical density (OD) of both C. albicansstrains were taken at 600 nm and recorded as 0.10.

The specimens were placed on Eliza plates and 1.5 mL of Yeast Extract Peptone (YPD) was added to each well. Next, 30µL of candida culture was added to the wells. Then, the wells were incubated at 37°C at 80 rpm for 24 h. At the end of 24 h, 1.5 mL of YPD was drained and 1.5 mL of new YPD was added to each well. The wells were then incubated again at 37°C at 80 rpm for 24 h.

The OD 600 nm spectrum was measured from the control group without C. albicans (0), the oral isolate of C. albicans strains (2.586), and the C. albicans ATCC 60193 strains (2.326) after 48 h.

The wells were washed twice with 2 mL of sterile water. After washing, the OD was measured at 600 nm from the oral isolate of C. albicans (0.036) and the C. albicans ATCC 60193 (0.050) strains.

Disinfectants were added to the Eliza plates. Thus, 2.5 mL of white vinegar was added to the vinegar group and left for 10 min. For the Corega group, Corega solution was prepared according to the instructions for use, and 2.5 mL of it was added to the Eliza plates and left for 5 min. Moreover, for the Saver group, 2.5 mL of chlorhexidine digluconate was added to the Eliza plates and left for 10 min. Finally, 2.5 mL of sterile water was added to the control group and left for 10 min.For all the groups, the disinfectants were drained and replaced with 2 mL of sterile water and kept at 100 rpm for 30 min. After vortexing, the specimens were transferred to Eppendorf tubes by making 10-3dilution. Then, 0.1 mL of the liquid was taken and inoculated into pads containing YPD medium. The pads were incubated for 48 h in an oven at 37°C. After incubation, the candida colony growth was measured on the pads. SEM images were taken from each group using randomly selected specimens.The data were analyzed by means of one-way ANOVA. The mean values and standard deviations of the groups were calculated.

3. Results and discussions

Statistically significant differences *(P<0.05)* were found between the three disinfecting method groups and the control group for the three denture materials according to both the Candida albicansATCC 60193 and oral isolate of Candida albicans strains (Table 1).

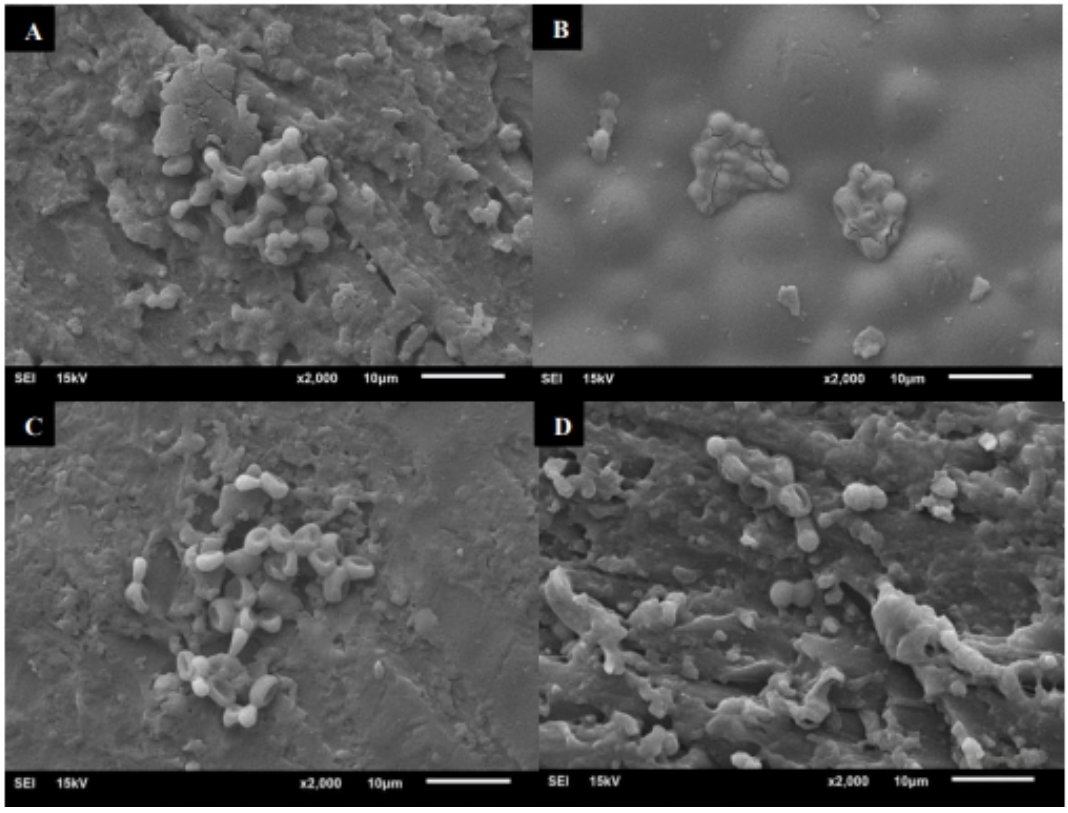
Table 1. Statistical analysis of antifungal effects

Materials	C. albicans strains	Methods	Mean	Std. Deviation	P
Autopolymerized acrylic resin	ATCC 60193	Control White vinegar Chlorhexidine digluconate Corega	30.33 4.83 0.00 5.16	25.48 2.85 0.00 5.11	0.0015
	Oral	Control White vinegar Chlorhexidine digluconate Corega	31.00 6.33 0.00 2.33	21.86 4.17 0.00 1.63	0.003
Heat.cured acrylic resin	ATCC 60193	Control White vinegar Chlorhexidine digluconate Corega	28.00 6.33 0.00 8.00	19.55 2.94 0.00 4.97	0.010

	Oral	Control	20.00	14.38	0.007
		White vinegar	4.50	3.50	
		Chlorhexidine digluconate	0.00	0.00	
		Corega	2.83	3.54	
Hard relining denture material	*ATCC 60193*	Control	64.50	20.76	0.000
		White vinegar	19.00	7.37	
		Chlorhexidine digluconate	0.00	0.00	
		Corega	9.50	10.78	
	Oral	Control	23.66	7.63	0.007
		White vinegar	19.16	15.94	
		Chlorhexidine digluconate	0.00	0.00	
		Corega	2.33	2.80	

Saver (2% chlorhexidine gluconate) was more effective than Corega or 100% white vinegar for Candida albicans ATCC 60193 and the oral isolate of Candida albicans for the autopolymerized acrylic resin, heat-cured acrylic resin, and hard relining material.No statistically significant differences *(P<0.05)* were found between the final counts of Candida albicans ATCC 60193 and oral isolate of Candida albicans after disinfection with 100% white vinegar and denture cleaning tablets (Corega). However, for the hard relining material, the amount of oral isolate Candida albicans was found to be statistically significant *(P<0.05)* after disinfection with 100% white vinegar and denture cleaning tablets (Corega).

The SEM images shows Candida biofilm on the denture base materials (Figure 2, Figure 5).



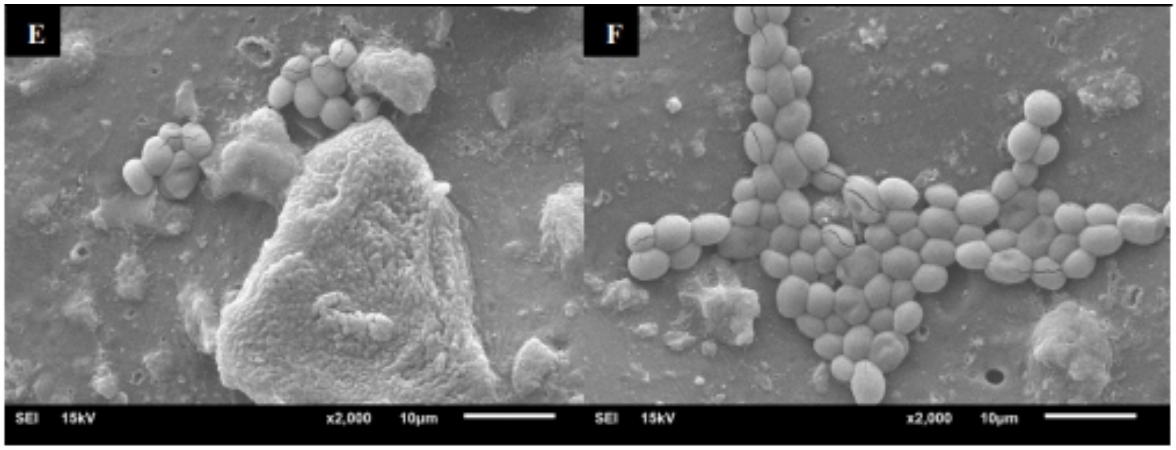
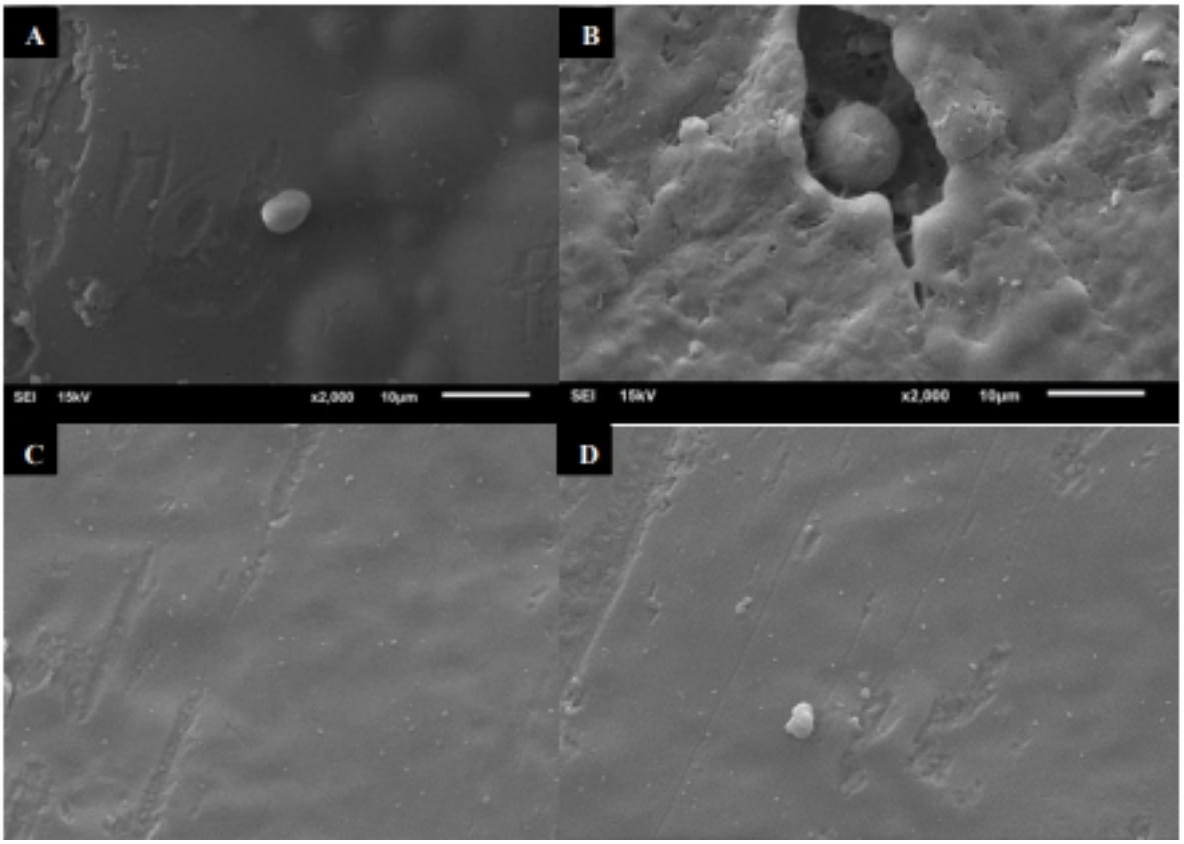


Figure 2. SEM images of the Control autopolymerized acrylic resin group (A) C. albicans ATCC 60193 and (B) Oral isolate of C. albicans. SEM images of the Control heat-cured acrylic resin group (C) C. albicans ATCC 60193 and (D) Oral isolate of C. albicans. SEM images of the Control hard relining denture material group (E) C. albicans ATCC 60193 and (F) Oral isolate of C. albicans



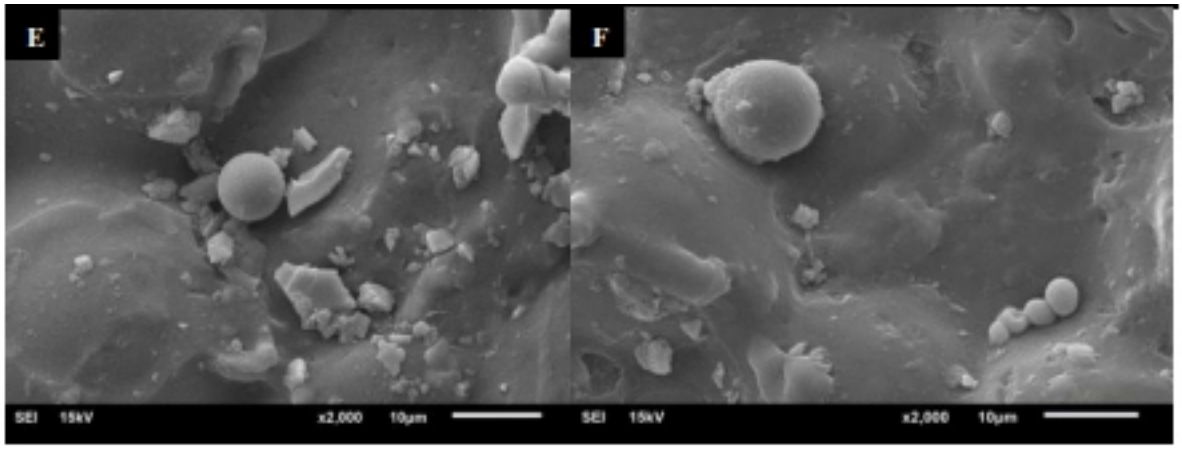


Figure 3. SEM images of the 100% white vinegar autopolymerized acrylic resin group (A) C. albicans ATCC 60193 and (B) Oral isolate of C. albicans. SEM images of the 100% white vinegar heat-cured acrylic resin group (C) C. albicans ATCC 60193 and (D) Oral isolate of C. albicans. SEM images of the 100% white vinegar hard relining denture material group (E) C. albicans ATCC 60193 and (F) Oral isolate of C. albicans

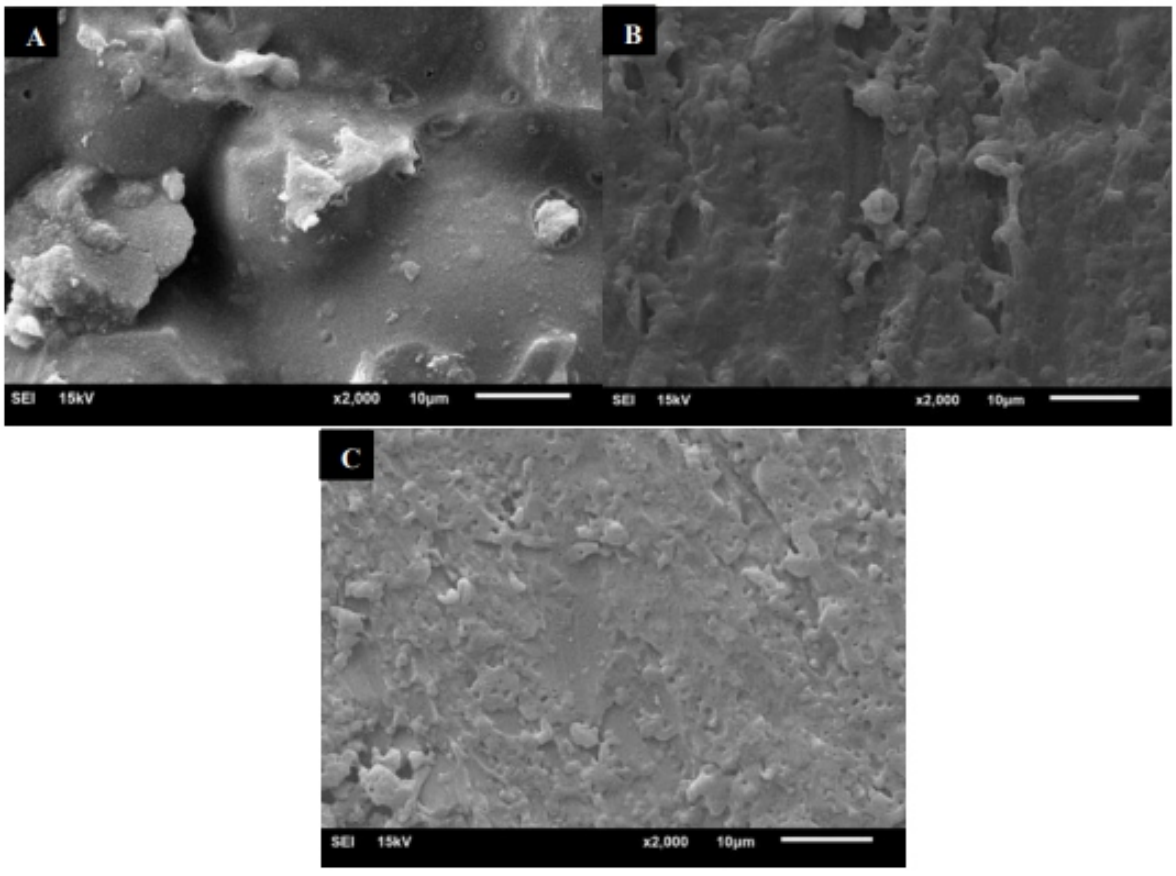


Figure 4. SEM images of the chlorhexidine digluconate (Saver) group (A) autopolymerized acrylic resin, (B) heat-cured acrylic resin, (C) hard relining denture material

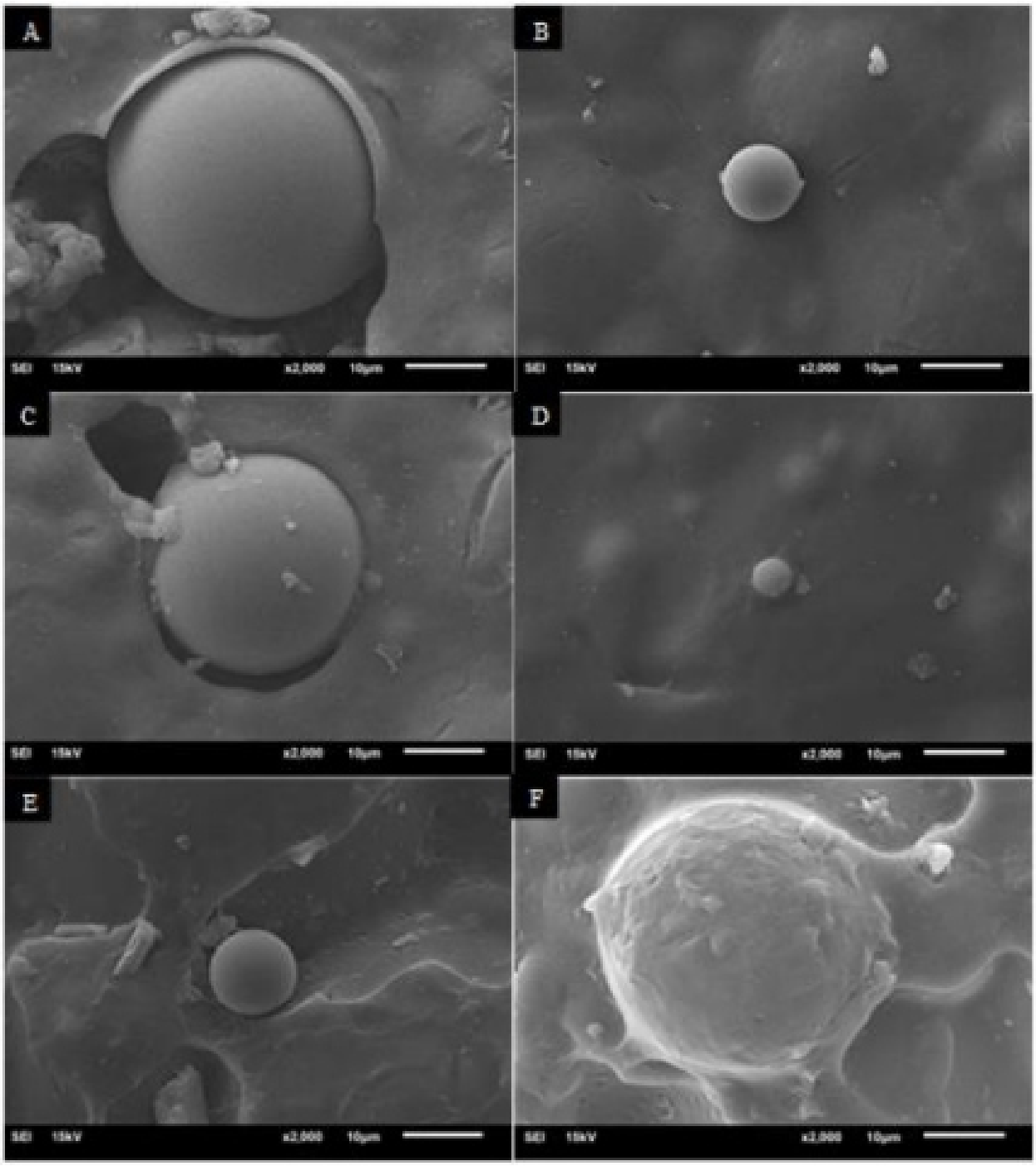


Figure 5. SEM images of the denture cleaning tablets (Corega) autopolymerized acrylic resin group (A) C. albicans ATCC 60193 and (B) Oral isolate of C. albicans. SEM images of the denture cleaning tablets (Corega) heat-cured acrylic resin group (C) C. albicans ATCC 60193 and (D) Oral isolate of C. albicans. SEM images of the denture cleaning tablets (Corega) hard relining denture material group (E) C. albicans ATCC 60193 and (F) Oral isolate of C. albicans

Routine cleaning is necessary to prevent denture stomatitis [13]. Chemical disinfectants are easier to use and more effective than mechanical cleaning [14]. The 2% chlorhexidine digluconate (Saver) solution was found to be successful, so the null hypothesis was supported.Vinegar is nontoxic; it is also an affordable household product. Yildirim-Bicer et al. [15] reported that use of 100% vinegar for 10

min effectively reduced C. albicans. In vitro studies have shown that the low doses of acetic acid in vinegar induce programmed cell death in C. albicans [16]. In the present study, the effect of white vinegar was significant on blocking and reducing the growth of colonies of C. albicans.

CHX 0.2% has been used to treat Candida-associated dental illness [17]. Pavarina et al. [18] immersed complete dentures in a 4% CHX solution for 10 min, which was effective in elimination of the C. albicans. Da Silva et al. [19] advocated the use of chlorhexidine digluconate (Saver) and showed that is highly effective against C. albicans, S. mutans, and S. aureus. In the present study, CHX (Saver) was found to be very efficient for preventing the growth of C. albicans.

Denture cleaning tablets (Corega) help remove biofilm and stains [20, 21]. De Freitas Fernandes et al. [22] showed that denture cleaning tablets were effective in reducing C. albicans strains. In the present study, the effect of denture cleaning tablets was found to be similar to that of white vinegar.

4. Conclusions

The effect of different disinfectant methods on reducing the growth of C. albicans was found to be significant. The 2% chlorhexidine digluconate (Saver) solution was found to be the most effective on C. albicans when different disinfectant methods were evaluated. The effect of the denture cleaning tablets (Corega) and 100% white vinegar was found to be similar.

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