Chemical treatment of acrylic teeth ridge lap area trough tensile strength investigations

Adelina Elena Stoia, Sorin Lakatos, Mircea Pielmusi, Cosmin Sinescu, Adrian Gheorghe Podoleanu, Mihai Rominu,

Abstract—The purpose of this study was associated to the surface treatments effect evaluation of three different organic solvents on the bond strength of artificial acrylic teeth to denture base repair resin. 40 large size acrylic molars (Spofa Dental complete denture kit) were used to make 40 acrylic cylinders. The milled cylinders were randomly assigned to four experimental groups, ten cylinders per each group. The cylinders flat surfaces were considered bonding areas.

The surface treatment regimens were: Group 1: polishing (control group), Group 2: ethylene chloride treatment; Group 3: ethyl acetate treatment; Group 4: acetone treatment.

A self-cured denture base repair resin (Duracryl – Spofa Dental) was used for manufacturing the bonding test specimens, according to the ADA specification No. 15. Each specimen was stored for 30 days in distilled water and tensile tested at a speed of 1 mm/min. The mean values of the tensile bond strength test registered were statistically significant among groups, ranging from 13, 67 MPa (group 4, acetone) to 29, 14 MPa (group 2, ethylene chloride).

The chemical treatment of acrylic teeth ridge lap area with ethylene chloride, led to an enhanced bond strength of artificial teeth to the denture base resin reported to the control group, group 3 (ethyl acetate) and 4 (acetone), but has reached lower value levels compared to the ANSI/ADA Nr. 15 according to which tensile strength value must be, if statistically significant, 31 MPa

Keywords—acetone, acrylic teeth, chemical treatment, denture base resin, ethyl acetate, ethylene chloride tensile strength.

I. INTRODUCTION

The advantages of acrylic teeth is their ability to realize an adhesive bond to the denture base resin. Although the bonding seems to be satisfactory, clinical failures are frequently reported in practice [1–3], especially those pertaining to frontal group, (Fig.1), (Fig.2).

Fig.1. Acrylic tooth detached from denture base resin

Fig.2. Acrylic tooth detached from denture base resin: detail.

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Previous studies have demonstrated that debonding of teeth from the base resin is the most frequent repair in practice, (Fig.3), (Fig.4), (Fig.5) the bond between acrylic denture teeth and denture base materials being unpredictable [2-5]. Previous studies report that 30% of denture repairs are due to debonded teeth [6-7], situation that generates stress and additional costs for the patients [6].

It is now known that a large variety of factors can generate the failure at the acrylic tooth–denture base resin interface. The failure between artificial acrylic tooth and denture base resin has causes such as wax residues on denture teeth ridge lap area [6], tin-foil substitute contamination [6–9], and different laboratory processing steps [9–11], the type of tooth material (conventional acrylic teeth or cross-linked teeth) [14,18], different or polymerizing processing methods applied to the base resins [2,19].

The wide variety of new materials, the different types of denture base resins and different type of artificial teeth materials and the variety of processing methods is responsible for a large variability of the results.

In the chemical structure of both acrylic teeth and self cured denture base resin, linear or cross linked polymers are to be found, among which methylmethacrylate, poly(methylmethacrylate), and other different monomers and additives.

Acrylate and methacrylate esters are derivatives of the corresponding acids, the respective structures are: CH$_2$=CHCOOR and CH$_2$=C(CH$_3$)COOR. The properties of acrylic ester polymers are dependent to a large degree on the type of alcohol from which the acrylate or methacrylate esters are prepared. Many of the physical properties and a few of chemical properties of the polyacrylates and polymethacrylates can be related to the glass-transition temperature and can be influenced also by the molecular weight of the polymer. Acrylic and methacrylic acids are, together with the crotonic acid, the simplest members of the family of $\alpha,\beta$-unsaturated carboxylic acids. The monomeric acrylic acid, CH$_2$=CHCOOH was prepared through the oxidation process of acrolein in 1843, and his polymerization ability was observed in 1872. The methacrylic acid CH$_2$=C(CH$_3$)-COOH was for the first time prepared from the roman camomile oil in the year 1879 and his polymerization ability was observed in 1893. The polycracylates obtained by a head-to-tail addition process consist of a hydrocarbon backbone with a pendant ester group. The polymethacrylates also have a pendant methyl group at the same carbon atom.

Polymethylmethacrylate (PMMA) in his pure form transmits extensively in the ultraviolet range so that commercial grades of PMMA are usually supplied with an added ultraviolet-radiation absorber which absorbs the strongest erythemal band of sunlight protecting in this way against sunburn. PMMA, like all organic plastics is hygroscopic, but the absorption-desorption process is almost perfectly reversible. Usually , PMMA, stored under ordinary atmospheric conditions, contains almost 0.5% water vapor. The polymers are the main component of the teeth and denture base resin. Polymers do not behave as ideal elastic materials in the solid state. The time and temperature dependent viscoelastic behavior of polymers determines the material properties which are important for the design of plastic components. The polymers are the components that determine their technological and physical behavior, which are characterized by long chains of repeated monomer units. The linear or branched macromolecules of plastics can be synthesized either by addition polymerization (the monomers are linked together without elimination of water or other simple molecules) either by condensation polymerization (monomers are linked together with the elimination of water or other simple molecules) The most used process for the synthesis of the polymers (thermoplastic) is double-bond polymerization. Among double bond polymerization and polycondensation, rearrangement polymerization known as polyaddition is another type of joining the monomers or intermediate products with different reactive end groups without formation of volatile products. This process is suitable for atmospheric pressure curing of plastics.

![Fig.3. Completes denture debonded acrylic tooth repair aspects.](image)

![Fig.4. Completes denture debonded acrylic tooth repair aspects: reattached tooth, oral view.](image)
Plastic denture teeth are made essentially of polymethylmethacrylate copolymerized with a cross linking agent as, for example, glycol dimethacrylate. It is known the fact that strongly cross-linked polymers are insoluble in organic solvents and also the fact that no polymer can be volatilized without chemical decomposition.

Solvents are liquids, solids, or even gas that are capable to dissolve other solids, liquids, or gaseous solute, resulting solutions that are soluble in a certain volume of solvent at a specified temperature. Most organic solvents have a lower density than water, which means that they are lighter and will form a separate layer on top of water. An important exception is represented by the most part of the halogenated solvents among which dichloromethane or chloroform. The organic solvents used in this study were acetone, ethylene chloride and ethyl acetate.

The large variability of results increases the need for further examination techniques in order to improve the bond strength of acrylic teeth to denture base resins.

Standards and test method standards have become very important in recent years, because many test methods use arbitrary conditions and procedures. For that reason, technologists have supported the development of standard procedures and the use of these in product specifications. A test method standard must be able to specify all the device parameters, the test piece details, the steps in the procedure and the presentation of the results.

The ultimate state of unity would be for all countries to use the same standards. This, obviously, will be of great importance making it much more easier for the technologists in order to exchange technical information. Also it would be a big step forward if all the countries could compromise on their national procedures in order to overcome the difficulties of language in an area where the language is the most important tool. A very important need for interlaboratory testing will be to produce precision statements for standard test methods.

Quality control or quality assurance is concerned with maintaining the quality of products. The selection of the actual tests used for control of the quality must be a compromise between scientific interest and economic necessity.

The American Dental Association (A.D.A.) specification No.15 defines standards regarding artificial resin teeth, the minimum bond strength required between artificial teeth and denture base resins.

The aim of this study is not focused on the development of new dental laboratory technologies regarding the repair of the detached teeth from the denture base resin. The study aim involves only the investigation of the chemical treatment effect induced by few organic solvents (ethylene chloride, ethyl acetate and acetone) to the acrylic teeth ridge lap area, in order to improve their bond strength to denture base resin. The study aim involves only the investigation of the chemical treatment effect induced by few organic solvents (ethylene chloride, ethyl acetate and acetone) to the acrylic teeth ridge lap area, in order to improve their bond strength to denture base resin. From the literature and also from praxis it is known that many attempts have been made to improve the bonding at the interface of acrylic teeth and denture base resin among which: mechanical treatments (Fig.5) [4,7,12,13], chemical treatments (the treatment of the tooth ridge lap area with monomer, organic solvents, or a combination of these [7,10,14]), treatments that have been reported efficient by some researches [12,15–17] and inefficient by others [6,10,13].

![Fig.5. Mechanical treatment of acrylic tooth ridge lap area.](image)

### II. MATERIALS AND METHODS

#### A. Sample Preparation

The samples were realized according to ANSI/ADA Specification Nr. 15.

Acrylic teeth and also self cured denture base resin belong to plastic materials family which has here complicated characteristics and behavior necessitating their own test procedures. In order to establish the bond strength between chemical treated artificial acrylic teeth and self cured denture base resin the tensile test was elected. Tensile strength is known to be the maximum tensile stress exhibited during a test, but occasionally is taken as the stress at break. The most common type of stress-strain measurement is made in tension, which is by stretching the material. A tensile stress is thus applied, defined for a section of uniform cross-sectional area $A_0$ by the formula $A_1 = F_1/A$ where:

- $A_1 = \text{tensile stress}$
- $F_1 = \text{tensile force}$

As the material starches so that his dimensions orthogonal to the axis of applied force decrease and for this reason the area of cross-section decreases. In order to realize experimental work, the most tensile strengths are based on the original cross-section ($A_0$) because this is much more easily to measure before the test has started. Elongation at break, the so called ultimate elongation, is represented by $l_1$ where $l$ is the length at failure. Usually, the elongation at break is expressed as a percentage of the original length:

$$\text{elongation at break} = \frac{l_0 - l}{l_0} \times 100\text{ per cent.}$$

The statement of results as per unit thickness implies that the property is proportional to thickness, is clear that size or shape will influence the final result.

The precise size and shape of test pieces has his importance in order to obtain ideally results when materials must be compared. The dumb-bell shapes are the most frequently used for tensile testing, but the sharp so called shoulders would...
After the milling procedure of the 40 artificial acrylic large molars (Spor Dental) 40 cylinders with 5 mm height and 6 mm diameter were obtained. The milling steps were realized with the JMA Dakar Alejandro Altun SA milling keys device, a trepan bur with 6 mm internal diameter and a diamond disc, attached in the mandrels milling machine.

generate a lower breaking load, because the shape has also some undesirable stress raisers.

The molars with the axial faces parallel milled were clamped in to the jaws of the device so that the vertical movement of the trepan bur, attached to the Dakar mandrel, milled the lateral surface of the acrylic cylinder.

Some of the tensile test specimen manufacturing steps are captured in Fig. 9, Fig. 10., Fig.11., Fig. 12., Fig.13.
The 2 flat bases of each one of the acrylic cylinders were submitted to the chemical treatment as it follows: Group 1 no treatment, Group 2. Ethylene Chloride, Group 3 Ethyl Acetate, Group 4 Acetone.

Ethylene chloride, also known as 1,2-dichloroethane, is a chlorinated hydrocarbon, used often in order to produce vinyl chloride monomer (chloroethene), which is the major precursor for PVC production. It is a colourless liquid with a chloroform-like odour. 1,2-Dichloroethane is used generally as an intermediate for other organic chemical compounds and also used as a solvent. As a good apolar aprotic solvent, 1,2-dichloroethane is used as degreaser and paint remover. It forms azetropes with many other solvents, including water (b.p. 70.5°C) and other chlorocarbons. 1,2-Dichloroethane is toxic especially by inhalation due to its high vapor pressure, corrosive, highly flammable and carcinogenic.

Ethyl acetate is the ester of ethanol and acetic acid; it is manufactured on a large scale for use as a solvent. He is an organic compound with the formula (CH₃)₂CO. It is a colorless, mobile, flammable liquid, being the simplest of the ketones. Acetone is miscible with water, and is a good solvent for most plastics and synthetic fibers including those used in laboratory bottles made of polystyrene, polycarbonate and some types of polypropylene and it is used by the pharmaceutical industry and as a denaturation agent in denatured alcohol. In the laboratory, acetone is used as a polar aprotic solvent in a variety of organic reactions. Donor number and donor acceptor scale measures polarity in terms of how a solvent interacts with specific substances. Protonic solvents solvate negatively charged solutes through hydrogen bonding. Aprotic solvents such as acetone or dichloroethane (have large dipole moments responsible for the separation of partial positive and partial negative charges within the same molecule) solvate positively charged species via their negative dipole.

After the chemical treatment the samples of the 4 groups were submitted to time domain C Scan OCT investigations.

Optical coherence tomography (OCT) is a powerful and sensitive tool for characterization of optical properties and imaging of superficial tissue [21]. OCT can achieve micrometre depth resolution and allows accurate in vivo measurement of thickness, area and volume in the tissue. In OCT, the depth dimension is explored by scanning the optical path difference (OPD) between the object path and reference path in an interferometer illuminated by a low coherence source. The maximum interference signal is obtained for OPD = 0.

In OCT, the achievable depth resolution is given by the optical source linewidth and not by the numerical aperture of the lens, as is the case in confocal microscopy. For OPD values larger than the coherence length of the source used, the strength of the interference signal diminishes considerably. This explains the selection in depth of the OCT [22].

Most OCT systems reported to date (Time domain or Fourier domain) can acquire only longitudinal (B Scan) sections of biological targets.
using flying-spot raster scanning [23] or capturing a full-field image of the sample [24].

Following the C Scan OCT investigations the cylinders were each one placed in to the pattern moulds, the dough stage dumping of the denture base resin paste being the next step of the manufacturing procedure of the tensile test samples. The finalization of the denture base resin curing process was followed by the realization of the final shape of the tensile test sample in accordance with ANSI/ADA specification Nr.15. as it can be seen in Fig.12.

Fig.14. The tensile sample shape in accordance with ADA Nr.15

All the samples were tensile tested, using Multitest 5i (Mecmesin) at 1 mm/min speed, according to one of the nine test speeds of ISO 527: Speed A 1 mm/min ±50%. Tensile stress is known as the tensile force per unit area of the original cross-section within the gauge length supported by the test piece at a given moment. The standard unit is mega Pascal (MPa) = MN/m² (Mega Newton/metre²) = N/mm²

III. RESULTS

Fig.15. The superficial layer of acrylic teeth before acetone treatment in Time Domian C Scan OCT noninvasive investigations.

Fig.16. Acetone effect induced to the superficial layer of acrylic teeth after 180 seconds treatment.

The C scan OCT time domain investigations allowed capturing data imaging regarding the effect of the organic solvents used in this study to the superficial layer of the acrylic teeth submitted to chemical treatment as it can be seen in Fig.16.

Fig.17. Detaille of the tensile test sample after the adhesive fracture

The tensile test strength values at which the acrylic tooth denture base resin adhesive interface cracked as it can be seen in Fig. 17 were one by one captured and registered in tables (Table.1), (Table 2).

In Table.1 are captured and expressed in Newton the tensile strength values of the samples pertaining to the 4 groups of the study. In Table 2 are expressed in MegaPascal the tensile strength values of the samples pertaining to the 4 groups of the study.
Table 1. The registered tensile strength values (in Newtons).

<table>
<thead>
<tr>
<th>Grup 1</th>
<th>Grup 2</th>
<th>Grup 3</th>
<th>Grup 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>Ethylene Chloryde</td>
<td>Ethyl Acetate</td>
<td>Acetone</td>
</tr>
<tr>
<td>783.08 N</td>
<td>774.88 N</td>
<td>503.02 N</td>
<td>326.40 N</td>
</tr>
<tr>
<td>748.60 N</td>
<td>744.36 N</td>
<td>602.22 N</td>
<td>447.92 N</td>
</tr>
<tr>
<td>770.08 N</td>
<td>800.04 N</td>
<td>559.54 N</td>
<td>235.688 N</td>
</tr>
<tr>
<td>709.04 N</td>
<td>900.08 N</td>
<td>677.39 N</td>
<td>363.42 N</td>
</tr>
<tr>
<td>815.86 N</td>
<td>887.364 N</td>
<td>910.81 N</td>
<td>518.00 N</td>
</tr>
<tr>
<td>760.19 N</td>
<td>850.34 N</td>
<td>616.63 N</td>
<td>352.68 N</td>
</tr>
<tr>
<td>802.30 N</td>
<td>778.62 N</td>
<td>588.37 N</td>
<td>439.16 N</td>
</tr>
<tr>
<td>778.64 N</td>
<td>798.06 N</td>
<td>681.06 N</td>
<td>348.16 N</td>
</tr>
<tr>
<td>810.21 N</td>
<td>907.14 N</td>
<td>510.94 N</td>
<td>490.02 N</td>
</tr>
<tr>
<td>768.38 N</td>
<td>790.14 N</td>
<td>901.49 N</td>
<td>338.55 N</td>
</tr>
</tbody>
</table>

The Newton is the SI unit for force; it is equal to the amount of net force required to accelerate a mass of one kilogram at a rate of one meter per second squared. It was named after Isaac Newton in recognition of his work on classical mechanics.

The Pascal (symbol: Pa) is the SI derived unit of pressure, internal pressure, stress, Young's modulus and tensile strength. It is a measure of force per unit area, defined as one Newton per square meter.

Table 2. The registered tensile strength values (in MegaPascals).

<table>
<thead>
<tr>
<th>Grup 1</th>
<th>Grup 2</th>
<th>Grup 3</th>
<th>Grup 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>Ethylene Chloryde</td>
<td>Ethyl Acetate</td>
<td>Acetone</td>
</tr>
<tr>
<td>27.71 MPa</td>
<td>27.42 MPa</td>
<td>17.80 MPa</td>
<td>11.55 MPa</td>
</tr>
<tr>
<td>26.49 MPa</td>
<td>26.34 MPa</td>
<td>21.31 MPa</td>
<td>15.85 MPa</td>
</tr>
<tr>
<td>27.25 MPa</td>
<td>28.31 MPa</td>
<td>19.87 MPa</td>
<td>8.34 MPa</td>
</tr>
<tr>
<td>25.09 MPa</td>
<td>31.85 MPa</td>
<td>23.97 MPa</td>
<td>12.86 MPa</td>
</tr>
<tr>
<td>28.87 MPa</td>
<td>31.40 MPa</td>
<td>32.23 MPa</td>
<td>18.33 MPa</td>
</tr>
<tr>
<td>26.90 MPa</td>
<td>30.09 MPa</td>
<td>21.82 MPa</td>
<td>12.48 MPa</td>
</tr>
<tr>
<td>28.39 MPa</td>
<td>27.65 MPa</td>
<td>20.82 MPa</td>
<td>15.54 MPa</td>
</tr>
<tr>
<td>27.56 MPa</td>
<td>28.24 MPa</td>
<td>24.10 MPa</td>
<td>12.32 MPa</td>
</tr>
<tr>
<td>28.67 MPa</td>
<td>32.10 MPa</td>
<td>18.08 MPa</td>
<td>17.43 MPa</td>
</tr>
<tr>
<td>27.19 MPa</td>
<td>27.96 MPa</td>
<td>31.90 MPa</td>
<td>11.98 MPa</td>
</tr>
</tbody>
</table>
Table 3 Test Kolmogorov - Smirnov

<table>
<thead>
<tr>
<th>Variable</th>
<th>N</th>
<th>max D</th>
<th>p</th>
</tr>
</thead>
<tbody>
<tr>
<td>NPA</td>
<td>40</td>
<td>0.197462</td>
<td>p &lt; .001</td>
</tr>
</tbody>
</table>

Table 4. Non-parametric test post-hoc (Mann-Whitney U)

The null distribution of this statistic is calculated under the null hypothesis that the samples are drawn from the same distribution (in the two-sample case) or that the sample is drawn from the reference distribution (in the one-sample case). In each case, the distributions considered under the null hypothesis are continuous distributions but are otherwise unrestricted. The two-sample KS test is one of the most useful and general nonparametric methods for comparing two samples. In the special case of testing for normality of the distribution, samples are standardized and compared with a standard normal distribution.

The significant ANOVA result suggests rejecting the global null hypothesis according to which the means are the same across the groups being compared. Multiple comparison procedures are then used to determine which means differ. The Kruskal–Wallis test is the non-parametric alternative to ANOVA.

Table 5. Non-parametric test ANOVA (Kruskal-Wallis ANOVA)

Basically in statistics, the Kolmogorov–Smirnov is a nonparametric test used for the equality of continuous, one-dimensional probability distributions that can be utilized to compare a sample with a reference probability distribution (one-sample K-S test), or to compare two samples (two-sample K-S test). This K-S test quantifies a distance between the empirical distribution function of the sample and the cumulative distribution function of the reference distribution, or between the empirical distribution functions of two samples.

IV. DISCUSSIONS

After a simple analysis of the Table 3 it can be observed that comparing the p-values significant differences were observed comparing Group 2, Ethylene Chloride with Group 3 Ethyl Acetate, and also with Group 4 Acetone., and even comparing the Group 3 with Group 4, and also with Group 1 (Control_no treatment). Between Group 5 and Group 1 also significant differences were observed.
Softening or swelling of the plastics polymeric substrate by a certain type of solvent is one of the steps of the adhesion mechanism, secondary forces between interwoven polymer chains are generating the bonding of the materials together. The swelling and softening of the superficial layer of the plastic polymeric substrate are followed in some solvents case by the hardening of the superficial layer of the polymeric sample, as it can be seen in Fig. 16, figure which captures with the help of time domain C Scan OCT aspects regarding the effect of acetone induced to the superficial layer of acrylic teeth after 60 seconds treatment time. Analysing the capture shown in Fig. 16 it can be seen that the superficial layer of the sample is similar to a white strip line, that could indicate in the hardening of the superficial layer of acrylic teeth sample. This hardening which affects the superficial layer of the acrylic teeth treated with acetone could be the reason of the lowest tensile test values recorded in this study as it can be seen in Table 1.

Voyutskii [20] developed the diffusion theory of adhesion of polymeric materials. The ethylene chloride treatment enhanced the bond strength between acrylic teeth and denture base resin compared to ethyl acetate and acetone but reported to the control group the results were statistically insignificant. The bond strength values reached by group 3 ethyl acetate and group 4 acetone were not high enough to be in complete accordance with the ANSI/ADA specification Nr.15 according to which the tensile strength between acrylic teeth and denture base resin must reach the value of 31 MPa.

Table 6 captures the graphic representation of the first tensile strength values (category 1), second tensile strength values (category 2), third tensile strength values (category 3), fort (category 4), fifth (category 5), sixth (category 6), seventh (category 7), eighth (category 8), ninth (category 9) tenth tensile strength values of all the 4 groups submitted to this study. The OY axis is used in order to represent the MPa values, from 0 MPa to 35 MPa.

Table 6. Graphic representation of the tensile strength samples values of the fourth groups of this study.

![Graphical representation of tensile strength values](image)

V. CONCLUSIONS

Within limitations of this study related to the research methodology and considering the fact that the tensile test values of ethylene chloride group are significantly higher than those of group 3 and group 4 but statistically insignificant reported to control group the following conclusion can be born

A. Ethyl acetate and acetone have not improved the bond strength of acrylic teeth to denture base resin

B. The ethylene chloride treatment does not enhance significantly the bond strength of acrylic teeth to denture base resin compared to control group

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