# Tensile testing, a method used to demonstrate the effect of organic solvents on acrylic teeth denture base resin bond strength

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*Abstract*— The evaluation of the artificial acrylic teeth denture base repair resin bond strength in tensile represents the main reason of this study.
Large size artificial acrylic molars were used to mille 50 acrylic cylinders. The cylinders were randomly assigned in five experimental groups, ten cylinders each. The bonding areas represented by the flat surfaces of the cylinders were submitted to a different treatment. Group 1: polished (control group), Group 2: polished+methyl methacrylate, Group 3: sandblasting + methyl methacrylate, Group 4: sandblasting+universal repairing adhesive (Clearfil Repair-Kuraray), Group 5: polished+dichlormethane. The sandblasting procedures were realized using 50 µm alumina, 30 seconds, from 10 mm distance. 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 then tested in tensile at a speed of 1 mm/min. The mean values of the tensile bond strength test registered were statistically significant among groups, ranging from 13,5 MPa (group 4) to 35,9 MPa ( group 5). Dichlormethane treatment enhanced bond strength to the artificial teeth, for this reason could be considered as a laboratory and clinical procedure in order to improve the quality of bonding.

***Keywords***—acrylic teeth, chemical treatment, denture base resin, dicloromethane, tensile strength

## I. Introduction

Although the number of edentulous people has decreased, there are still many patients needing treatment that require complete dentures. The acrylic resin denture base material has been used in dentistry for more than 50 years. Acrylic resin was introduced on the market in 1937 and since then has enjoyed a large popularity, attributed to its simple processing technique and low cost of fabrication process. Even so, although materials with superior properties are available on the market and a large variability of fabrication techniques have been used to manufacture dentures, the acrylic resin still remains the most popular choice.

One of the primary advantages of acrylic teeth is their ability to adhesively bond to the denture base resins. Previous studies have demonstrated that although the bonding seems satisfactory, failures are still common so that detachment of teeth from the base resin is the most frequent repair in the laboratory praxis for conventional phrostodontics. This detachment may be attributed to a lesser ridge lap surface area available for bonding and the direction of the stresses encountered during function. The bond strength of different denture teeth to their denture bases can be in some situations high enough to cause tooth fracture without detachment and if the bond between the two main parts, teeth and denture base resin resists until the materials fail, it can be considered that the bond has fulfilled its functional requirements. However, bond failures between artificial acrylic teeth and denture base resins can occur and remain a major problem in complete dentures prosthodontic praxis, the bond remaining unreliable, inconsistent and unpredictable.

Complete dentures acrylic teeth detachment, (Fig. 1), (Fig. 2) even if it does not generates a physical suffering similar to the loss of a natural teeth, surly, from the psychological point of view, could be considered a tragedy for the patient, whatever his age or social position are. Acrylic teeth adhesion to denture base resin generates the longevity of the complete denture, for this reason the acrylic tooth becomes part of the whole: the complete denture. The detachment of acrylic teeth from complete denture bases, especially those that restore the complete denture frontal area, achieves values between 20%-30%.[1], [2] so from this point of view, the most common reason for the elderly group of patients of the population to seek dental treatment is for the replacement of missing teeth.

Fig. 1. Denture base acrylic tooth detachment, buccal aspect

Fig. 2 Denture base acrylic teeth detachment, incisal aspect

The main directions of investigation of the interfaces between artificial teeth and denture base resin were aimed at determining the factors that are generating negative or positive influences to the adherence of the teeth to the denture base, factors such as: 1. Teeth and denture base resin manufacturing technology, 2. Factors involved in the laboratory technological steps of samples manufacturing : wax impurities [3]. or gypsum impurities [4], 3. Physical or chemical ridge lap area treatment agents (such as organic solvents, curing agents, monomers adhesives) [5] [6] [7] [8]. 4. The action time of physical and chemical agents on the acrylic tooth ridge lap area; 5. Technological methods for dough stage denture base acrylic resin preparation (the amount of monomer and polymer in respecting the manufacturers indications) 6. Acrylic resin denture base polymerization method (auto polymerization, heat polymerization, baropolymerization, microwave polymerization) [9] [10] [11]. Last but not least in terms of importance, some of the factors that may change the adhesion of acrylic teeth to denture base resin, occur after the samples were made, namely water storage parameters.

Many attempts have been made to improve the bonding at the interface of acrylic teeth and denture base resin including mechanical and chemical treatment of the artificial teeth so called ridge-lap area. The ridge lap area is in fact the flat basal surface of the acrylic tooth.

The variability of ridge lap area treatments and results increases the need for further examination techniques in order to improve the bond strength between acrylic teeth and denture base repair materials.

Successful denture repair is based on the phenomenon of adhesion. Strong bonding of the surfaces improves the strength of the repaired unit and reduces stress concentration. Adhesion between denture base and repair materials can be improved by applying compatible chemicals to the acrylic resin surfaces. These chemicals etch the surface by changing morphology and chemical properties of the materials. Normally this change is obtained in the dental laboratories by wetting the surfaces with methyl methacrylate. Organic solvents such as methylene chloride have also been used for this process. It was reported that these organic solvents increase the bond strength of a repair material to the denture base.

The present paper does not aim to describe the technological aspects of the daily commonly known laboratory procedures regarding acrylic teeth reattachment to denture base resin. (Fig. 3).

Fig. 3.Acrylic teeth denture base resin reatachments aspects

## II. MATERIALS AND METHODS

The null hypothesis is based on the idea that physical or chemical treatment of the "ridge lap area" does not improve the adhesion of acrylic teeth to denture base resin.

The samples were made so that their material, size and design to subscribe ADA specification No. 15.

### *A. As a first step*

50 artificial acrylic first upper and lower molars (Spofa Dental) were used for milling 6 mm diameter base and 5 mm height cylinders.

This method uses a keys milling device, JMA Dakar, Alexandro Altun, SA which allows milling in perpendicular planes.

In order to generate the 6 mm diameter and lateral surface of the cylinder, a 6 mm internal diameter trepan bur was mounted in the mandrels milling machine.

After the trepan bur was fixed to the mandrels JMA Dakar, and the artificial molars with the axial sides milled as parallel planes were clamped in to the jaws of the machine, the movement in vertical plane of the bur, at a minimum length of 7 mm inside the molars, under cooling water jet realized the lateral surface of the cylinder (Fig. 4).

Maintaining the artificial molars clamped in the same position to the jaws of the machine and replacing the trepan bur with a diamond disc (Fig. 5), and moving it in a horizontal plane, perpendicular to the cervico-oclusal axis of the molars in mesio-distal direction, at minimum 1 mm distance below the mucosal surface of the acrylic teeth, the first base of the cylinder was made.

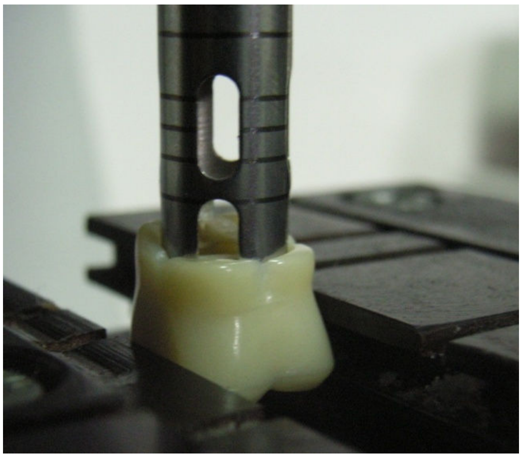

Fig. 4 JMA Dakar device (clamping jaws, trepan bur, acrylic tooth).

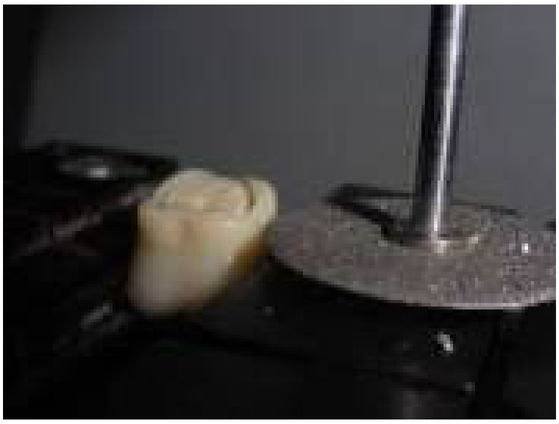

Fig. 5 Acrylic molar flat milled ridge lap area

The acrylic tooth with the milled lateral surfaces of the cylinder was removed from the clamping jaws of Dakar JMA. That allowed the removal of the acrylic tooth axial walls surrounding the lateral surface of the with a cylindrical shaped bur. A solid cylindrical shape with 6 mm height and diameter, a flat base , the other base being still represented by the oclusal surface, was obtained (Fig. 6).
The cylindrical solid shape is fixed again in the clamping jaws of the milling machine, this time with the oclusal surface directed to the disc fixed in the Dakar s JMA mandrel.
Moving the disc in mesio-distal way in a plane perpendicular to the cervico-oclusal axis of the cylindrical solid shaped body, to a predetermined length of 5 mm from the previously obtained, the second base of the cylinder was made ( Fig. 7).
The final shape corresponds to a cylinder with a diameter of 6 mm and length 5 mm, subscribing the ANSI/ADA No. 15 (Fig. 8).

Fig. 6 The lateral surface of the cylinder

Fig. 7 The flatt milling of the second base of the cylinder

Fig. 8. The final shape of the milled cylinders.

*B. The second step*

of the sample manufacturing, involves wax models preparation for the extremities of the samples.
The silicone putty impression of a mettalic object generated the wax sample. The metalic object corresponds in shape and size to a half wax sample. (Fig. 9a). After casting, solidification and removal of the wax from the silicone putty

impression, half of the wax samples were obtained, wax sample dimensions being equal to those of the imprinted metallic object (Fig. 9b). By bonding two wax half models at the 6 mm diameter bases, a wax model of a whole sample was made at the size specifications set by ANSI/ADA No.15

Fig. Nr. 9a. The metallic object.

Fig. Nr.9b. Wax model of the metallic object

### C. *In the third step*

patterns for future samples were made. 10 mould patterns suitable in size and design for the proper alignment of the 5 wax samples were used. Class four (IV) gypsum was chosen for the pattern manufacturing stage (Fig. 10). The manufacturing of the pattern involved the alignment in horizontal position of the wax models after gypsum paste preparation, so that the bases of the cylinders to be parallel with the bases of the extremities of each .

Fig. Nr.10. Wax samples removed from plaster models.

### D. *The next step*

refers to the treatment, either chemical or micromechanical of the two flat bases resulted after the milling procedure of the acrylic molars. The acrylic cylinders were divided into five groups, so that 10 cylinders are part of each one of the five groups. The flat surfaces, actually the two bases of the cylinders were considered as bonding areas. The surface treatment regimens were different from one group to the other: Group 1: polished (control group), Group 2: polished + methylmethacrylate, Group 3: sandblasting (Fig. 11) + methylmethacrylate, Group 4: sandblasting + universal repairing adhesive (Clearfil Repair-Kuraray), Group 5: polished+dichlormethane.

All sandblasting procedures were performed using 50 µm alumina, also known as aluminum oxide ( 30 seconds), from a distance of 10 mm.

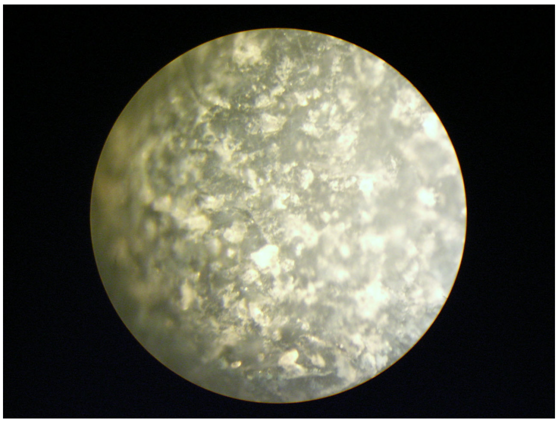

Fig. 11. 100X optical microscopy capture of 50 µm $Al_2O_3$ particule embedded in the acrylic teeth ridge lap area after sandblasting procedure

Aluminum oxide is a inorganic compounds with the chemical formula $Al_2O_3$. It is an amphoteric oxide and is commonly referred to as alumina, corundum as well as other names, reflecting its occurrence in nature and industry. Its most significant use is in the production of aluminum metal, although it is also used as an abrasive due to its hardness and as a refractory material due to its high melting point. As an abrasive aluminum oxide is used for its hardness and strength. It is used as a coarse or fine abrasive, including as a much less expensive substitute for industrial diamond. Different types of sandpaper use aluminum oxide crystals. In addition, its low heat retention and low specific heat make it widely used in grinding operations, particularly cutoff tools. As the powdery abrasive mineral aloxit, it is a major component, along with silica, of the cue tip "chalk". Aluminum oxide powder is used also in compact discs polishing and scratch-repair kits. Its polishing qualities are also behind its use in toothpaste. Alumina can be grown as a coating on aluminum by plasma electrolytic oxidation. Both its strength and abrasive characteristics are due to aluminum oxide's great hardness , he reaches the position 9 on the Mohs scale of mineral hardness. In dentistry, aluminum oxide is used as a polishing agent to remove stains. It is an alternative to sodium bicarbonate, for patients that have high blood pressure.

After the flat surfaces treatment of the 50 cylinders belonging to this study was realized, each cylinder belonging to the 5 groups was placed one by one in the middle of each of the five patterns of a mould, so that the bases obtained after cylinder milling to be located at equal distances from the extremities of the patterns (Fig. 12).

Fig. 12. Cylinders aligned in the mould patterns.

*E. The final step*

consisted in preparation and mould stamping of self cured acrylic denture base resin in the dough stage phase (Duracryl SPOFA Plus Dental, Kerr Company). The polymerization process followed in accordance to the manufacturer's directions. (Fig. 13).
After completion of polymerization and unpacking, (Fig. 14) the samples were kept in distilled water for 30 days at a temperature of 37 degrees Celsius (Fig. 15)
Subsequently, the samples were tensile tested, using Multitest 5i (Mecmesin) at a speed of 1 mm / min (Fig. 16).
Fig. 16 depicts the adhesive fracture at the interface acrylic tooth denture base resin after the tensile test was conducted and completed with the recording of the strength values at witch the acrylic tooth denture base resin adhesive interface cracked.

Fig. 13 Acrylic resin dough stage tamping in the mould patterns.

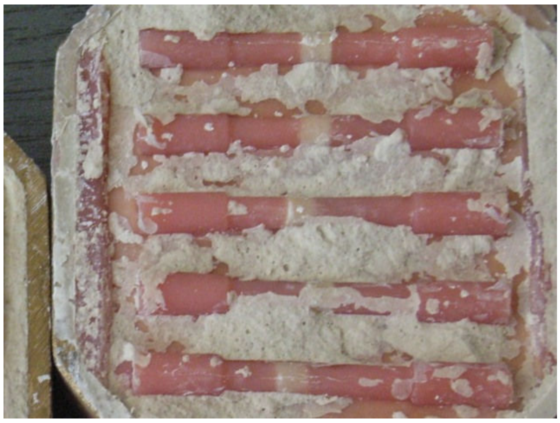

Fig. 14. Sample unpacking aspects

Fig. 15. The shape and size of the sample corresponding to specification ADA / ANSI No.15.

## III. RESULTS

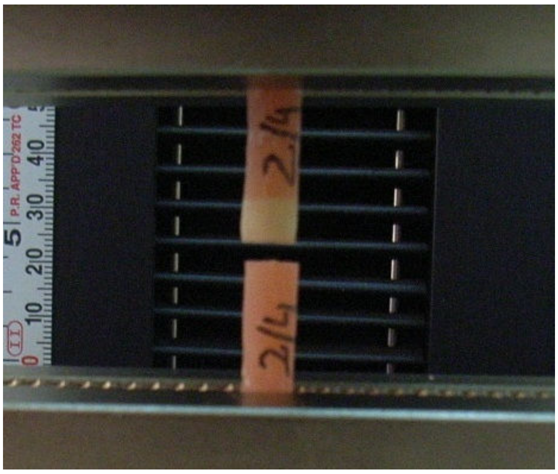


Fig. 16. Sample fixed to the Mecmesin holding device before after the adhesive fracture

Tensile strength values to which one of the interfaces gives up are presented in Table I and in Table II are obtained by the formula:
$R = F / S$,
where F = force and S = surface.

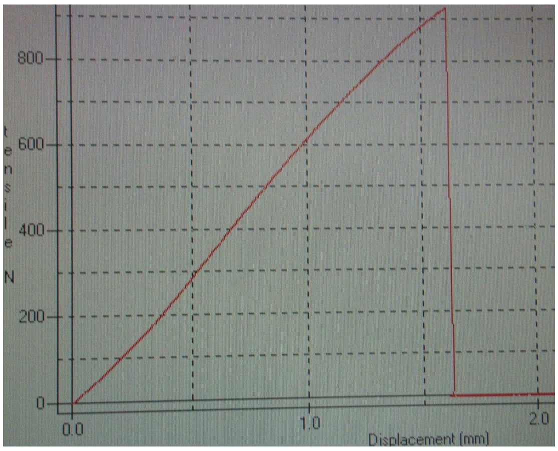


Fig. 17.Capture of the graphic depicting a 900N force responsible for adhesive fracture of one of the specimens.

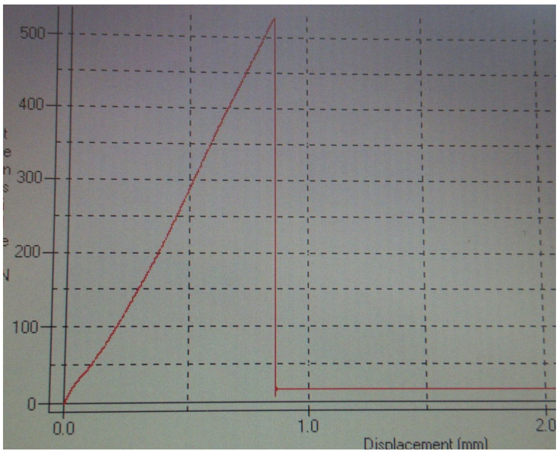


Fig. 18. Capture of the graphic depicting a 500N force responsible for adhesive fracture of one of the specimens.

After statistical analysis of results (One-Way ANOVA) significant differences were found between group five and four, insignificant differences between group one, two and three, and significant differences between group five and group one. One-Way Analysis of Variance is a way to test the equality of three or more means at one time by using variances based on the assumptions that all the populations from which the samples were obtained must be normally or approximately normally distributed, the samples must be independent and the variances of the populations must be equal. The null hypothesis of the One-Way ANOVA will be that all population means are equal, the alternative hypothesis is that at least one mean is different.

Table I: Tensile strength values in Newtons.

| | Group I Control | Group II | Group III | Group IV | Group V |
|---|---|---|---|---|---|
| 1 | F=783,2 N | F=830,4 N | F=842,7 N | F=315,1 N | F=1000,6 N |
| 2 | F=748,8 N | F=759,5 N | F=791,2 N | F=476,2 N | F=928,9 N |
| 3 | F=770,3 N | F=845,9 N | F=870,8 N | F=379,9 N | F=1016,7 N |
| 4 | F=709,1 N | F=839,9 N | F=754,0 N | F=328,8 N | F=998,4 N |
| 5 | F=816,4 N | F=882,1 N | F=753,4 N | F=431,2 N | F=1015,2 N |
| 6 | F=760,3 N | F=840,1 N | F=790,3 N | F=360,4 N | F=996,3 N |
| 7 | F=802,4 N | F=870,3 N | F=850,6 N | F=390,3 N | F=1004,6 N |
| 8 | F=778,9 N | F=860,9 N | F=810,4 N | F=410,8 N | F=970,4 N |
| 9 | F=810,3 N | F=865,3 N | F=781,2 N | F=351,9 N | F=1009,3 N |
| 10 | F=768,6 N | F=834,4 N | F=820,3 N | F=524,4 N | F=987,4 N |

Table II: Tensile strength values in MegaPascals

| | Control Group I | Group II | Group III | Group IV | Group V |
|---|---|---|---|---|---|
| 1 | 27,7MPa | 29,3 MPa | 29,8 MPa | 11,1 MPa | 35,4 MPa |
| 2 | 26,4 MPa | 26,8 MPa | 27,9 MPa | 16,8 MPa | 32,8 MPa |
| 3 | 27,2 Mpa | 30,2 MPa | 30,8 MPa | 13,4 MPa | 35,9 MPa |
| 4 | 25,0 MPa | 29,7 MPa | 26,6 MPa | 11,6 MPa | 35,3 MPa |
| 5 | 28,8 MPa | 31,2 MPa | 26,6 MPa | 14,6 MPa | 35,9 MPa |
| 6 | 26,9 MPa | 29,7 MPa | 27,9 MPa | 12,7 MPa | 35,2 MPa |
| 7 | 28,3MPa | 30,7 MPa | 30,0 MPa | 13,8 MPa | 35,5 MPa |
| 8 | 27,5 MPa | 30,4 MPa | 28,6 MPa | 14,5 MPa | 34,3MPa |
| 9 | 28,6 MPa | 30,6 MPa | 29,7 MPa | 12,4 MPa | 35,7 MPa |
| 10 | 27,1 MPa | 29,5 MPa | 29,0 MPa | 15,0 MPa | 34,9 MPa |

Table III: Statistical analysis of results using One-Way ANOVA.

| | Sum of Squares | df | Mean Square | F | Sig. |
|---|---|---|---|---|---|
| **Between Groups** | 2561,07 | 4 | 640,27 | 370,71 | ,000$^s$ |
| **Within Groups** | 77,72 | 45 | 1,73 | | Within Groups |

In order to compare two by two the five groups, the option Post Hoc multiple comparisons, ANOVA test, was chosen, as follows:

Multiple Comparisons - Post Hoc "Scheffe" Test.

Significant differences between the five groups, with $\alpha$= 0.001.

Legend: s = significant differences
ns = not significant differences

Only groups I, II compared with III give insignificant differences.

Table IV: Statistical analysis of results using Post Hoc "Scheffe" Test.

| (I) GROUP | (J) GROUP | Sig. | IV. SIG.LEVEL ($\alpha$) |
|---|---|---|---|
| control | **polished + MMA** | ,004$^s$ | **0.01** |
| | **sandblasting + MMA** | ,285$^{ns}$ | **0.05** |
| | **sandblasting + Kuraray** | ,000$^s$ | **0.001** |
| | **polished + $CH_2Cl_2$** | ,000 $^s$ | **0.001** |
| polished + MMA | **sandblasting + MMA** | ,468 $^{ns}$ | **0.05** |
| | **sandblasting + Kuraray** | ,000$^s$ | **0.001** |
| | **polished + $CH_2Cl_2$** | ,000$^s$ | **0.001** |
| sandblasted + MMA | **sandblasting + Kuraray** | ,000$^s$ | **0.001** |
| | **polished + $CH_2Cl_2$** | ,000$^s$ | **0.001** |
| sandblasted + kuraray | **polished + $CH_2Cl_2$** | ,000$^s$ | **0.001** |

## IV. DISCUSSION

The present study demonstrates that the different treatment of the acrylic teeth ridge lap area generates differences more or less significant in terms of acrylic resin denture base acrylic teeth tensile strength, differences that are in direct causal relationship with the type of treatment.

The results of this study showed that the tensile strength values are significantly different between group I (control) and group II (polished + MMA (methyl methacrylate)) ($\alpha$ = 0.01), the group II (polished + MMA (methyl meethacrylate)) being associated to higher values of tensile strength than group I (control). Methyl methacrylate is an organic compound with the formula $CH_2$=$C(CH_3)COOCH_3$. It is a colorless liquid, the methyl ester of methacrylic acid (MAA), a monomer produced on a large scale for the production of poly(methyl methacrylate) (PMMA). Methyl methacrylate is also used for the production of the co-polymer methyl methacrylate-butadiene-styrene (MBS), used as a modifier for PVC. The explanation of the result obtained in the group II of this study could be the one chosen by[12]. According to this the MMA (methylmethacrylate) treatment dissolves the PMMA (polymethyl methacrylate) structure and improves the adhesion between acrylic teeth and self cured acrylic denture base resin. Authors such as[13]-[14] found that, after there following studies, methyl methacrylate improves adhesion of acrylic teeth to denture bases, while, authors such as [15] support lower values of adhesion after methyl methacrylate treatment.

Comparing the control group (I) to the group V (polished + dichloromethane) it was found that the values of group V are significantly higher than those of the group I ($\alpha$ = 0.001). Dichloromethane (DCM, methylene chloride) is an organic compound. His chemical formula is $CH_2Cl_2$. It is a colorless, volatile liquid with a light sweet aroma and is widely used as a solvent. Although it is not miscible with water, it is miscible with many organic solvents. It was first prepared in 1840 by the french chemist Henri Victor Regnault. He exposed a mixture of chloromethane and chlorine to sunlight and isolated from this mixture dichloromethane. Dichloromethane's volatility and ability to dissolve a range of organic compounds makes him a useful solvent for many chemical processes. Concerns regarding its health effects have led to a search for alternatives in many of these applications Dichloromethane chemically welds some types of plastics; for this reason beeing used to seal the casing of electric meters. Often is sold as a dominant component of plastic welding adhesives, and it is also used at widely in the model-making industry in order to join plastic components together. Dichloromethane has the lowest toxicity of all the simple chlorohydrocarbons, but it is not without its health risks because its high volatility makes him a real dengerous acute inhalation hazard. Dichloromethane is metabolized by the body to carbon monoxide, fact that could potentially lead to carbon monoxide poisoning. Acute exposure by inhalation has degenerated in optic neuropathy and hepatitis. Skin contact during a long period of time can be associated with the ability of dichloromethane to dissolve some of the fatty tissues in skin, resulting in skin irritation or chemical burns. It may be carcinogenic, as it has been linked to cancer of the lungs, liver, and pancreas in laboratory animals. Dichloromethane crosses the placenta. Fetal toxicity in women who are exposed to it during pregnancy, has not been proven. In animal experiments, it was fetotoxic at doses that were maternally toxic. Even so teratogenic effects were not seen. In many countries, products containing dichloromethane must carry labels warning of its health risks. In the European Union, the European Parliament voted in 2009 to ban the use of dichloromethane in paint-strippers for consumers and many professionals.

Dichloromethane is a volatile organic solvent that applied to the ridge lap area of the acrylic teeth dissolves the superficial layer of the prefabricated high cross-linked polymer network, penetrating through polymer chains, expanding them, creating in this manner the premises to the presence of spaces between the polymer chains were MMA could penetrate. High values of tensile strength of acrylic teeth and denture base resin are obtained and explained by[16] based on the softening and "penetration" capacity of the solvent in the PMMA layer, practical the ability to achieve a new polymer intertwined network. Authors such as[17]-[18] have found an improvement in adhesion after treatment with dichloromethane

Lowest values of tensile strength were recorded in the Group IV (micro sandblasted + adhesive Kuraray) .All these low values can be explained by the complex mechanism of adhesion. The low efficiency of $Al_2O_3$ microsandblasting associated with the Clearfil Kuraray adhesive chemichal treatment could find an explanation by the type of the monomer from the adhesive system , monomer represented by 10-methacrylate-oil-dihydrogen-phosphate-oxidecil. His monomer has a molecular structure represented by a hydrophobic $(CH_2)_{10}$ chain at whose extremities could be found a methacrylate group and a hydrophilic phosphate group represented by the radical O = P-(OH) responsible for performing a chemical bond between bivalent $Ca^{2+}$ ions from the enamel structure and also with the bivalent ions from the composition of alloys used in prosthetic restorations. The polymeric structure of the acrylic teeth does not offer the potential to make new chemical bonds with 10-methacrylate-oil-oxidecil-dihydrogen-phosphate, fact which could explain the low values of adhesion for the group IV.

Reporting the group II (polished + MMA) to the group III (sandblasted + MMA) significant differences were found between the values of tensile strength of the two groups(=0.05). These facts indicate that $Al_2O_3$ micro sandblasting associated to methyl methacrylate treatment do not improves significantly the adhesion of acrylic teeth to the denture base resin.

Within the limitations of this study related to the research methodology the increased adhesion of acrylic teeth treated with dichloromethane to denture base resin was demonstrated.

## V. CONCLUSIONS

A. Dichloromethane significantly improves the adhesion of acrylic teeth to denture base resin, tensile strength values recorded in the group V (polished + dichloromethane), being significantly higher than the amount stipulated by the ANSI/ADANr.15 (31 MPa), the acrylic teeth ridge lap area treatment with dichloromethane being considered as a leading treatment in order to improve the bond strength of artificial acrylic teeth to denture base repair resin..

B. Microsandblasting associated to MMA treatment do not cause statistically significant superior results compared to polishing.

C. The adhesive system Clearfil Kuraray is not indicated for complete denture repairs .

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