

# The Role of Functional Polymers in the Optimization of the Acrylic Biomaterials Used in Removable Prosthetic Restoration

## III. Behaviour of the adhesive used for the multilayer consolidation

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*The paper represents a third part from the series, and refers to the elaboration of the experimental protocol regarding the use of Iosipescu test in the optimization of the structural-constructive characteristics of the matrix biomaterials for removable prosthetic restoration. The way the samples were consolidated with aluminum plates, at the clamping ends, was established through traction and shearing tests. Based on the theoretical protocol, the following were determined: the shape, size and loading of the Iosipescu sample, alongside with the bridge calibration. The gathered experimental data were used to evaluate the behavior of the CT-1 silicon adhesive. The paper focuses on the design of the samples used for the traction and shearing tests, made out of biocompatible polymer composite materials. Their structural and feasible behavior is influenced by the adhesion between layers, and by the gluing capacity of the used adhesive. The purpose of the paper is to enhance the performance of polymer composite materials, by using a compatible adhesive in creating a finite modeling element for standard single traction and shearing resistance tests. A tridimensional model of a longitudinal sample that underwent stretching and shearing test, while applying quasi centric loads, was elaborated and the main characteristics of the adhesive and of the glued interfacial area, that influence the structural and functional performance, were analyzed.*

**Keywords:** Iosipescu test, silicon resin, poly (methyl methacrylate), removable prostheses, traction resistance, modulus of elasticity, Poisson's coefficient, adhesive bond line, finite element model

The constructive optimization of the matrix biomaterials used by the dental medicine in removable prosthesis takes into consideration their structural and functional characteristics that are determined through a series of mechanical traction, bending, compression, torsion and shearing resistance tests [1-4]. The most often tested are the resistances to traction and shearing, through the Iosipescu test [5, 6].

The accurate knowledge of the mechanical properties of prosthesis, made of polymeric composites, is a fundamental requirement for its utilization as a competitive structural biomaterials.

The mechanical tests are the only way to determine the stress-strain response of biomaterial [7].

During the last decades, an intensive research effort has been devoted to the experimental identification of shear properties of anisotropic polymeric composite materials. Different shear test methods have been proposed, among which are the Iosipescu shear test and the off-axis tensile test [8].

The Iosipescu shear test was first developed for measuring the shear strength of metal rods [1, 2, 7], and has been studied extensively by the composite research community within the last 20 years, starting with the work of Walrath and Adams [9] in the early eighties. The fixture developed by Adams and Walrath [10], known as the 'modified Wyoming fixture, was included in an ASTM Standard (D 5379-93) [11] and is widely used in composite research laboratories. An alternative fixture design was proposed by Pierron [12-15], in order to overcome a certain number of drawbacks of the modified Wyoming fixture [16].

Due to the fact that the structural behaviour and the feasibility of the polymer materials used in removable prosthetics in dental medicine depend both on their nature and physical-structural characteristics and those of the binding adhesive, the design of the Iosipescu samples takes into consideration the architecture (the layers arrangement and their geometry) of the areas glued with adhesive [17, 18].

In order to evaluate the performance of the polymer components and of the adhesive involved in fixing the composite structures, a model sample has to be created and after evaluated through Iosipescu traction and shearing test [1, 2, 17, 18].

The direct modeling of different properties of some polymer materials, like orthotropic elasticity and slow deformation, are described by Konnerth et al [17], and Kaliske and Rothert [18].

Often, different available software are used to create a sample to be tested through Iosipescu test, due to the great number of modeling variants, especially when using synthetic textile polymer materials, natural or metallic insertions and different adhesives. The model sample for this type of tests must contain a low number of adjacent layers, glued together with adhesives [19-22].

The purpose of the paper is to make easier the Iosipescu test, highlighting the behaviour of the adhesive in multilayer polymer structures for removable prosthesis. Taking into consideration previous results of the authors [1-2] regarding the influence of different parameters on the mechanical load used in tractions and shearing tests, this research uses also a simple tridimensional model, which allows for an easy comparison of the results, correlated with the used

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materials and provides a set of elementary data for further research regarding the adhesive influence on the tensions that can appear in removable prosthetics.

## Experimental part

### Materials and method

#### The design of the Iosipescu samples

The classic composite testing samples are parallelepiped (with a rectangular section), axially symmetric and with a middle-shaped nick V, both on the upper and lower side (fig. 1). On the area clamped in jaws, the polymer samples are consolidated with aluminum plates glued with silicon resin. The clamping jaws have a differential arrangement:

- for traction, the jaws are set up to the limit, close to the V nicks, under the action of the force  $F$  (fig. 1a);

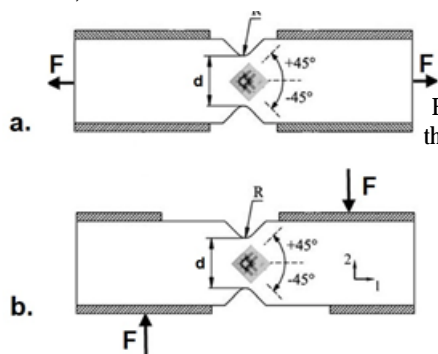


Fig. 1. The shape of the classic Iosipescu test samples [1]:

a - traction;  
b - shearing;

- for shearing, one jaw is set on one side where the  $F$  force acts, up to the limit of the V nick, and on the other side, opposite to the  $F$  force, only up to the half (fig. 1b).

In order to transform the applied load into traction or shearing force, devices with adequate clamping systems were used to fix the metal head of the samples. The systems act on the minimum resistance middle area of the sample, right on the V nicks (classic samples).

During the traction test, the samples were under the influence of the pulling forces. The test used a dynamometer like device, that can determine the longitudinal elasticity module ( $E$ ) and the *Poisson coefficient*, these being two of the most important characteristics for bi dimensional mathematic modeling of different structures of permanent or removable prosthetics, evaluating the tension transmitted to the two essential components of the prosthetic field: the bone and the covering mucus membrane.

In return, the classic samples found under the action of the shear force are done like the traction ones, with specially conceived devices. As mentioned before, the samples have the heads reinforced with aluminum plates and the central narrow area shaped as a V, on which the tensometric transducer used to measure the deformation was glued [16]. The measuring device comprises a classical rosette, type CEA-06-062WT-120, made by Micromeritics (USA), with two tensometric transducers at a  $90^\circ$ . These resistant tensometric transducers used to measure the sample response to the shearing force (the rosette is composed from two resistive electric transducers, TER, with grills oriented at  $\pm 45^\circ$  angle towards the shearing section). They measure the specific linear deformations: TER1 measures  $\varepsilon + 45^\circ$ , of compression and TER 2 measures  $\varepsilon - 45^\circ$ , of traction.

The signal from the transducer is amplified by a N2324 tensometric bridge, integrated in a data acquisition system.

The force applied on the samples was calculated almost up to the breaking point, and the speed with which the load was applied (lower than 3N/s) ensured the deformation compensation and allowed for the accurate

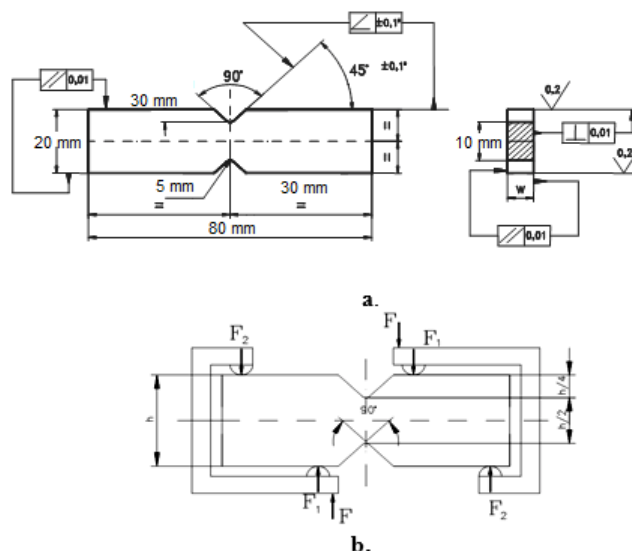


Fig. 2. The shape and dimensions of classic Iosipescu samples, used in the two tests [1]: a. The dimensions of the classic sample used in the traction test and the execution requirements;

b. The dimensions of the classic sample used in the shearing test and the execution requirements

use of the machine (stopping the machine on time to analyze the continuous deformation).

Figure 2 presents the shape, dimensions and theoretical load of the Iosipescu sample

During the experiment, in order to evaluate the adhesive's behaviour in composite structures during shearing and traction tests, two classic Iosipescu samples were used: a simple one, without adhesive (with a single plate,  $80 \times 20 \times 5$  mm), a second one, with two plates ( $80 \times 20 \times 2.25$ ), glued together with adhesive, inserted with polyethylene fiber, thinned in the V center, perpendicular on the adhesive interface and an experimental model with a structural architecture with three glued plates, without textile insertion, to allow the evaluation of the adhesive behaviour.

The design of this sample (this composite experimental model) took into consideration a well known tridimensional model [23, 24], often used in Iosipescu tests, for traction and shearing resistance to quasi centric loads, in order to properly evaluate the behaviour of the CT-1 adhesive silicon used in gluing polymer materials for removable prosthetics in dental medicine. The sample geometry and architectural layering were done accordingly with EN 302 Standard (2004) [25]. In order to improve the local resolution in the breaking area, the following system was used: a central PMMP plate ( $80 \times 20 \times 2$  mm), on which, at the end of both sides, two other plates (one of  $30 \times 20 \times 1.50$  mm and the other of  $40 \times 20 \times 1.50$  mm) glued in zigzag with adhesive silicon.

On the other side of, two other plates with the same dimensions were glued in an opposite manner (fig. 4). This type of geometry is compatible with the optimal surface on which the adhesive was added, equal on both sides of the central piece.

The base characteristics of this composite sample involved in the traction and shearing tests differ a lot from those of the classic Iosipescu sample. In this specific case, these characteristics are: the thickness of the central plate ( $d \times g$ ), the area of the glued surfaces and the geometry of the breaking area.

Initially, in order to optimize the constructive characteristics of the experimental model composite sample, the most important parameters, that influence the sample's behaviour (the area of the glued surfaces in the

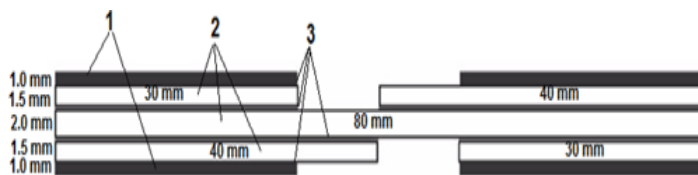
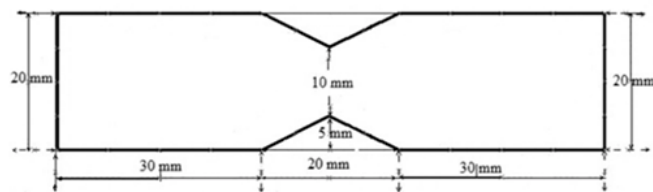
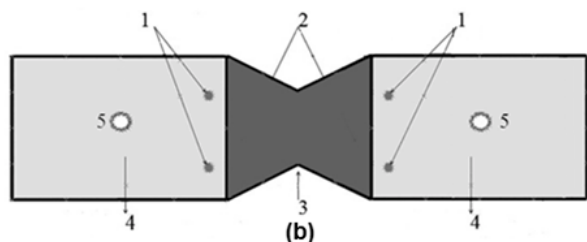


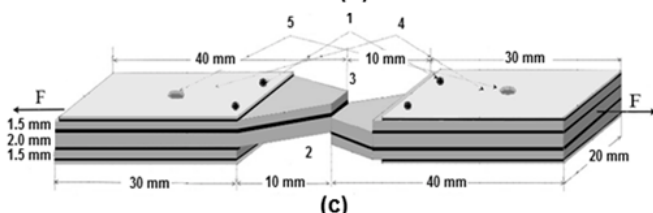
Fig. 3. The dimensions of the experimental model sample in composite structure: 1 – The aluminum plated surface that will be fixed into the testing device; 2 – The PMMP plates used to create the composite structure; 3 – The CT-1 adhesive silicon pellicle



(a)



(b)



(c)

composite structure of the sample, the area of the surfaces glued with aluminum plates in order to be fixed in the jaws of the testing device, the thickness of the adhesive layer, the area of the free surfaces and the area of the breaking section), were varied.

Numerical investigation of the traction and sheering tests were done to examine and improve the applied tested method and to analyze the tensions that appear in glued areas under the variation of the relevant parameters.

During the standard test the lateral side of the specimen was observed by a distortion-free CCD camera (charged-coupled device). The resulting image sequence was then analyzed with a cross-correlation algorithm in the software VIC 2D (Correlated Solutions, USA). It was decided that the thermal IR analyses should be done right before commencing the breaking process of the sample exposed to the traction tests.

Figure 3 presents the dimensions of the experimental model sample used in traction and sheering Iosipescu type tests, in composite structure.

In order to ensure the precision of the tensometric measurements, the dimensions of the sheering sample and the execution indications followed to the ASTM D 5379-93 recommendations.

The load applied to the samples is the sheering force, obtained by applying asymmetrical forces to the vertical axis, with the help of an adequate device [26-35].

Based on relevant literature [1, 2, 18-35] on removable prosthetic materials based on acrylic polymers, simple and with various metallic or textile reinforcements, the study regarded methyl methacrylate, silicone rubber and CT-1 silicone adhesive. All chemicals were obtained from Sigma-Aldrich Chemical Co., Inc., Milwaukee, WI. The Poly (methyl methacrylate) strength properties during moulding differ significantly on its directions as a result of the orientation effect. This material tends to creep. It is not suitable for operation under multiple dynamic loads, this being the reason for using reinforcements made of

Fig. 4. The dimensions (a) and the final aspect (b) of the two classic samples (without adhesive and composite with adhesive) and for the experimental model composite sample with adhesive (c) for the Iosipescu test:

1 – points used to connect the sample to the tensometric measuring device; 2 – polymeric sample; 3 – the area exposed to the mechanical load (traction and sheering), with the rosette with tensometric transducers used to measure the Iosipescu sample response to the sheering test; the area that is clamped in to the device, with aluminum plates reinforcements; 5 - clamping bolt hole

polymeric textiles materials [1, 2, 31-33]. In addition to these polymers, for the doping of poly (methyl methacrylate), denoted PMMA (Sigma-Aldrich Chemical Co., Inc., Milwaukee, WI). In this case, 2-5 mm thick PMMA plates were cast separately. These were adjusted by rotative grinding on abrasive surfaces until they became suitable as layered probe. The two plates were bonded with CT-1 silicone adhesive. Polyethylene fibers mesh reinforcement was placed between these plates. The reinforcement was immersed in silicone adhesive for bonding. The test specimens were molded from acrylic polymer in silicone rubber tanks. After hardening they were removed and mechanically processed to obtain the standard shapes and dimensions of the Iosipescu tests. Firstly, the study considered the optimization of aluminum mounting plates on the ends with silicone resin. This approach was tested using the shear test (fig. 4).

The experimental model sample was structurally optimized, based on the results previously obtained [1, 2], having the same dimensions like the witness sample (length 80.00mm, wide 20mm and thickness 5.00mm). On the classic samples, the V nicks are done perpendicularly on the middle of the samples, with a central depth of 5.00mm and with a distance between the V ends of 20.00mm.

During the sheering test, in the central of the area fixed into the device, a hole with  $f$  diameter was done, in order to fix the clamping bolt.

The deformation area of the experimental model sample, were the rosette with tensometric transducers is places, differs quite a lot from the classic samples. Two structural elements of the experimental model sample respond in a different manner to the two tests (traction and sheering): the central breaking section of the base plate and the glued side plates on its ends, which move under the load of the traction force and come off at a certain load of the sheering force.

## Results and discussions

A set of samples to be tested through Iosipescu test, was created using two plates of poly (methyl methacrylate), named PMMP, between which a polyethylene fiber net was inserted and fixed with CT-1 silicon adhesive. The same adhesive was also used to glue



Sample	Sample Composition		Dimensional characteristics				$F_{max}$	$\sigma_{max}$	$\sigma_{max}^{real}$	Traction behavior
			L	l	d	g				
	Base material	Reinforcement	mm	mm	mm	mm	N	MPa	MPa	Breaking characteristics
Witness	PMMA	Without reinforcement	80	20	10	5.00	1480	26.30	29.60	Transversal, under the clamping jaws
Classic model	PMMA	Polyethylene fiber net /silicon resin CT-1	80	20	10	2.25	2260	41.47	45.20	Eccentric, Longitudinal
Experimental model	PMMA	Without PMMA reinforcement/ silicon resin CT-1	80	20	10	2/2x1.5	820	37.80	41.00	Eccentric, Longitudinal

**Table 1**  
THE SAMPLES RESULTS ON THE TRACTION TEST

Sample	Glued surface	Breaking force	$\tau_{max}$ (conventional)
	mm <sup>2</sup>	N	MPa
Classic model, without adhesive (witness)	$2 \cdot 30 \cdot 20 - \pi \cdot (4.0)^2 / 4 = 1174.88$	1480	1.260
Classic composite model, with adhesive	$2 \cdot 30 \cdot 20 - \pi \cdot (4.0)^2 / 4 = 1174.88$	2260	1.924
Experimental model, with adhesive	$2 \cdot 30 \cdot 20 - \pi \cdot (4.0)^2 / 4 = 1174.88$	820	0.698

**Table 2**  
THE PARAMETRS OF THE SAMPLES EXPOSED TO THE SHEERING FORCE

the aluminum plates on the clamping ends of the samples in the jaws of the test device.

The traction and sheering tests were used to study the behavior of the adhesive used to create the composite sample, by gluing two polymeric plates and the reinforcement system, and to glue the aluminum plates on the clamping end of the samples.

This study was necessary to establish the architecture of the double layered samples and the dimensions of the surface on which the aluminum plates were applied, a practical aspect that influences the accuracy of the final results, for two samples: the witness sample (without insertion) and the experimental model sample (with insertion).

Table 1 present the structural composition of the three sample used in the Iosipescu test and the mechanical characteristics determined through the traction test, data then used to compare the two samples (the classic model and the experimental one).

The symbols used in table 1 are: L - samples length, l - samples width, d -sample width between the V nicks (at the middle), g - the thickness of a polymeric plate (PMMA),  $F_{max}$  - the maximum force, at which the breaking occurs,  $\sigma_{max}$  - the normal maximum tension ( $\sigma_{max} = F_{max} / S_0$ ), where  $S_0 = d \times g$  is the section where the breaking occurs (or the initial section of the sample study area),  $\sigma_{max}^{real}$  = the normal real maximum tension, calculated in the section were the breaking occurred, were the effect of traction load is often eccentric due to the asymmetrical structure and to the way the structures takes the load.

The values obtained for the normal maximum tension,  $\sigma_{max}$ , vary between 29.60MPa for the classic witness sample without reinforcement, 45.20MPa for the composite classic sample with reinforcement and drops to 41.00MPa for the experimental model sample. These differences are attributed to the presence or to the lack of the adhesive in the central area (with or without reinforcement), to the geometry of the area were the tensometric transducers are placed and to the layering composition of the samples.

The data from table 1 confirm the layering and insertions role on the mechanical traction resistance, on the

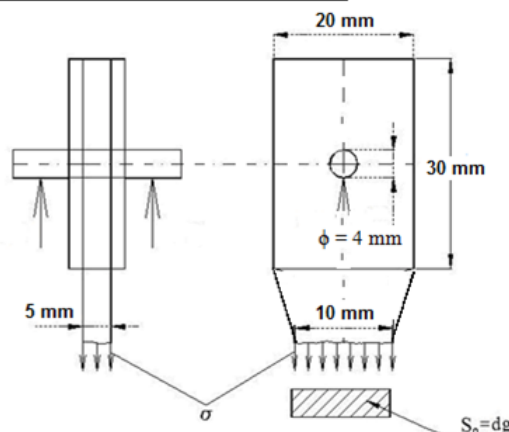


Fig. 5. The denotations used in the calculus of clamping head dimensions in relation to the shearing resistance of the adhesive used for the fixing of the aluminum plates for clamping jaw [1]

dimensional characteristics and on the structural composition of the specimens, correlating with  $F_{max}$  and  $\sigma_{max}$ .

Table 2 presents the behaviour of the three samples, under the load of the sheering force.

Analyzing the data from table 2, a variation of the tangential sheering force of the adhesive can be observed, from 1.260 MPa for the sample without adhesive to 1.924MPa, for classic composite model, with adhesive and to 0.698MPa, for the experimental model, with adhesive.

The dimension of the clamping heads was determined through calculations based on the traction resistance of the specimen  $\sigma_{max}$ , from where the maximum force was established:

$$F_{max} = S_0 \cdot \sigma_{max} \quad (1)$$

where  $S_0 = d \times g$

The tangential tension is calculated using the following relation (2):

$$\tau_{max} = \frac{F_{max}}{S_{forf}} = \frac{S_0 \cdot \sigma_{max}}{2 \left( 600 - \frac{\pi \phi^2}{4} \right)} \quad (2)$$

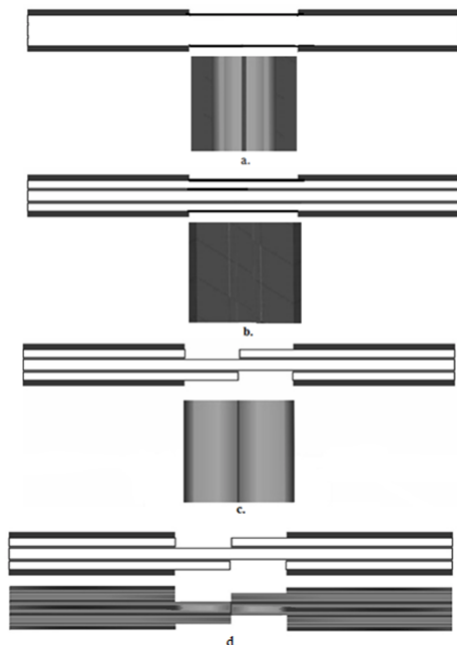


Fig. 6. Thermal IR image: a - Iosipescu classic sample of polymeric material PMMP; b - Iosipescu sample classic multilayer adhesive CT-1 impregnated textile insertion polyethylene fiber; c - Iosipescu experimental sample for evidence of adhesive behavior in multilayer structures of the side area (a-c) and in layer section (d)

Table 3  
SENSITIVITY ANALYSIS: EFFECT OF THE PARAMETER VARIATION ON THE MAXIMUM AND MINIMUM SHEAR STRESS  
VALUES AND THEIR RATIO

Parameter	Variation range (MPa)	Max value [MPa]	Min value [MPa]	Max/Min
Elasticity of adhesive	100...800	7.0-125.0	5.0-45.0	1.4-2.78
Elasticity of adherent	800...2500	20.0	16.0	1.25
Elasticity of interface zone	0.15...0.5×2260	5.5	5.2	1.058

were  $F_{max}$  is the maximum applied force, calculated according to the relation (1),  $S_{for}$  is the surface exposed to the sheering force, comprised of the two sides of the clamping head of the sample with a surface of 600mm<sup>2</sup>, out of which the campling blot hole dimension is extracted  $\pi\phi^2/4$ .

If the traction test is represented trough the  $\sigma_{max}$  obtained values, the sheering one is well represented through  $\tau_{max}$ , the two being very important characteristics used in correlation with the structure functional characteristics of the removable prosthetics.

Figure 6 presents the IR thermal image for the two classic samples, view from the side (a and b) and for the experimental model sample on the breaking point (c) and in layer section (d), all under the action of traction force load.

The thermal IR imaging done on the three samples exposed to traction forces shows differences only between the aluminum plate used on the clamping head of the samples (fig. 6A, b and c). In the transversal section (d), beside the gradient of the central plate, there are other thermal colors, differentiated on materials (PMMP polymer-gradient blue and the CT-1 silicon adhesive gradient red), the structural modifications being well noticeable (dynamic flows).

For a correct estimation of the variations determined trough the two traction and sheering test and their stress effect on the interfacial areas (glued areas of the PMMP plate with CT-1 adhesive), an analysis on the adhesive layer behavior was performed.

The elasticity and rigidity variations of the interface area and their effect on the traction and sheering stress are presented in table 3 (the variation domains of the elasticity of adhesive, elasticity of adherence, elasticity of interface zone), alongside with the max and low values of stress in the most exposed point from de adhesive area of the sample. The rapport between the max and the lowest

values of the three parameters was taken into discussion, as a measure of the sensitivity of the traction and sheering effect.

The elasticity variation is treated using the Young module variation of adhesive and glued area, depended on the thickness of the adhesive layer, after hardening. A variation in the rigidity of a fictive interface area has, none the less, a relative small influence on the stress of the glued bindings.

## Conclusions

The paper represents a third part from the series that uses the Iosipescu test to develop an experimental protocol for optimizing the structural constructive characteristics of some samples made from acrylic polymers with or without textile insertions and silicon adhesives, biocompatible with dental medicine removable prosthetics. The way the samples were consolidated with aluminum plates, at the clamping ends, was established through traction and sheering tests, in correlation with the sample dimensions.

The experiment used type CT-1 silicon adhesive, based on acrylic polymers, as such or in the form of double layered matrix system, with or without polyethylene fiber insertions.

Based on the obtained experimental data and theoretical evaluations, the following conclusions can be drawn:

-based on a theoretical protocol, two types of tests (traction and sheering) were selected from the Iosipescu test for removable matrix polymer prosthetics, in order to evaluate the adhesive behavior.

-two samples were used for the traction and sheering tests, a witness one, made put of poly (methyl methacrylate), PMMA and a second sample, made from two plates half as thick as the first one, with polyethylene fiber insertions, glued together with silicon adhesive CT-1,

also used to glue the aluminum plates on the clamping ends of the samples.

-The values obtained for the normal maximum traction value,  $\sigma_{\max}$ , vary between 29 and 45 MPa, and the tangent sheering tension of the adhesive, compared to the sample without adhesive,  $\tau_{\max}$ , drops from 1.260 MPa for the sample without adhesive to 1.924 MPa, for classic composite model, with adhesive and to 0.698 MPa, for the experimental model, with adhesive;

- both values vary accordingly to the dimensional characteristics and structural composition of the samples, thus confirming the role of multi layering, adhesive and insertions on the mechanical resistance to traction;

-analyzing the data from table 2, a variation of the tangential sheering force of the adhesive can be observed, the traction test ( $\sigma_{\max}$  values) and the sheering test ( $\tau_{\max}$ ) were necessary in order to establish the dimensions of the surface on which the clamping jaws of the device should be fixed, a practical aspect that influences the accuracy of the final results;

-comparative thermal IR analysis done from the side of the samples and layer structure of the samples at the breaking point allowed us to observe the unevenness of the gradient colors under the influence of the traction force, differentiated on two materials (PMMP polymer – gradient blue and CT-1 silicon adhesive gradient red), the structural modifications being well noticeable (dynamic flows).

-the elasticity and rigidity variations of the interface area and their effect on the traction and sheering stress are presented in table 3 (the variation domains of the elasticity of adhesive, elasticity of adherence, elasticity of interface zone), alongside with the max and low values of stress in the most exposed point from the adhesive area of the sample.

-the rapport between the max and the lowest values of the three parameters was taken into discussion, as a measure of the sensitivity of the traction and sheering effect.

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