The Role of Functional Polymers in the Optimisation of Acrylic Biomaterials used in Amovable Prosthetic Restoration

I. The experimental protocol using the Iosipescu test

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This paper is the first note on experimental protocol development regarding the use of losipescu test specimens with acrylic fibre insertion or without the insertion, with metal or textile fibres in a series of tests in which the structural and constructive characteristics of matrix biomaterials are optimised for removable prosthetic restoration. Tensile and shear tests allowed the determination of specimen consolidation with aluminium plates of clamping jaw heads. Based on the theoretical protocol, the following were determined: shape, size and loading of losipescu specimen and deck sizing. The CT-1 silicone adhesive behaviour was evaluated based on experimental data. Poly (methyl methacrylate), denoted as PMMA, was used in the study. PMMA has polyethylene fibre and copper wire mesh inserts. These were individually assembled using this adhesive that was used to fix aluminium plates on the clamping jaw heads. The values obtained for the maximum normal traction stress, σ_{max} ranged between 22 and 60 MPa, and the tangential shearing stress τ_{max} , of the adhesive ranged between 2 and 6 MPa, with an average value of 4.75 MPa, both depending on the dimensional characteristics and structural composition of the specimens, thus confirming the role of stratification and inserts in the mechanical strength of the specimens. Stress values of σ_{max} and σ_{max} allowed the determination of the dimensions of the surface the specimen clamping jaws are fixed upon, practical aspect influencing the accuracy of the final results.

Keywords: losipescu test, silicon resin, poly (methyl methacrylate), removable prostheses

The unbreakable bond between the mechanical strength and the nature/structure of biomaterials forming the removable prosthesis and the prosthetic field characteristics is a key element in achieving individual prosthesis [1, 2]. A key issue in the clinical purpose of removable prosthesis is the correlation between the types of biomaterials used, the technological line and the prosthetic field particularity of the edentulous elderly patient [3].

To this end, a series of contributions are known regarding the use of sandwich matrix systems based on polymers with fibre inserts and/or mesh reinforcements [4].

The optimization of these matrix biomaterials, which takes into consideration the structural and functional characteristics of removable prosthetics, involves a series of mechanical tests of material strength under tension, compression, bending, shear and torsion [5, 6]. Among these, the losipescu test is often used as a tension and shear test [7].

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

The mechanical tests are the only way to determine the stress–strain response of biomaterials [8].

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 losipescu shear test and the off-axis tensile test.

The Iosipescu shear test was first developed for measuring the shear strength of metal rods [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], in order to overcome a certain number of drawbacks of the 'modified Wyoming fixture.

The majority of works about the Iosipescu shear test addressed the problem of correct measurement of shear modulus and shear strength of synthetic composite materials. The first issue is more or less a solved problem [13], however the second one is far from being solved [14, 15]

The work is part of an extensive study representing the first note which considers the theoretical foundation of the experimental protocol regarding the two applications of the losipescu tensile and shear tests for polymer composite materials based on acrylate used in removable prosthesis and the final prosthesis.

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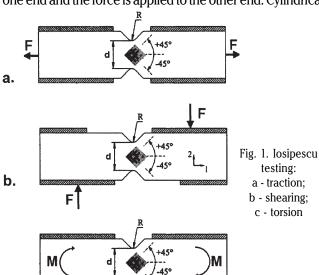
Experimental part

The Iosipescu experimental test protocol

Test *tensile*, *shear* and *torsion* tests were proposed by Nicholas Iosipescu following his investigations from 1960-1962 [7, 16]. Initially both the specimen and the method of Iosipescu for the first two tests were mainly used to study metal. Subsequently they have been extended to polymeric materials and composites at the Wyoming University [9, 10].

The Iosipescu test considers only three types of experimental approaches: the tensile, shear and torsion tests. For each test, the samples involved in the experiment are compositionally and dimensionally different depending on the the nature and structural formula of the composite material. The physical principle of operation of the test, the jaw/mandrel clamping mode and force distribution are also different. The test samples or test specimens are rectangular (with rectangular or square cross section), axially symmetrical and have a middle V shaped notch. both in the upper and the lower parts (fig. 1) or may have a cylindrical shape (circular cross section with a V-shaped bottleneck perimeter). In the testing device's fixing zone, the composite polymer specimens are reinforced with aluminium plates glued with silicone resins. The specimen clamping plates have a differential displacement: for the traction test, they are placed up to the clamping jaw, on both sides, near the V-shaped grooves, under the action of force F (fig. 1a); for the *shearing test* the plates are placed, one end, on the plane force F is acting upon, up to the Vshaped notch, and on the other end on the opposite plane force F is acting upon, up to the V-shaped notch (fig. 1b). In case of torsion (twisting) test, the plates are disposed on both ends up to the V-shaped notches (fig. 1c). The jaws grasp the entire surface of the specimen up to the V-shaped notches and force M is applied (torque).

Where three-dimensional mathematical modelling must be carried out for various stratified or unstratified structures with complex profiles, mechanical testing can be extended to other forms of testing: *compression* when the specimen is cylindrical or prismatic (without central notch) and is freely placed on the lower anvil of the device. The deformation force is applied by the upper anvil of the device. *Bending* is another test. The specimen may have any cross section, but with the same profile length. The specimen is freely placed on the device. Its ends are supported by bearings and the deformation force is applied in the middle section from top to bottom by a V-shaped anvil. In the latter case, the specimen can be embedded at one end and the force is applied to the other end. Cylindrical



or prismatic profiles are only bent once. Rectangular, flat, profiles are bent multiple times until they brake.

To transform the applied stress into a tensile one shearing or torsional stress, suitable devices are used. The metal ends of the sample are clamped with jaws, which act on the area of minimum weakness. This area is located in the middle section of the sample, the area with V-shaped indentations.

The traction samples are subject to pulling. These tests involve a dynamometer type device that allows the determination of the longitudinal elasticity modulus (E) and of the Poisson coefficient. These are two very important characteristics for two-dimensional mathematical modelling of various total prosthetic structures, evaluating the transmission of stress to two essential components of the prosthesis: bone and covering mucosa.

Shearing tests are carried out in a manner similar to traction tests, using specially designed devices. As noted, specimens have clamping jaws reinforced with aluminium plates and V-shaped bottleneck central area. A strain gauge measures the deformation [17]. The latter is a 06-062WT CEA-120 rosette, manufactured by Micromeasurements (USA) comprising two 90° oriented strain gauges. These resistive strain gauges used to measure the specimen response to shearing (the rosette is composed of two resistive electrical transducers, in short TER, with grids oriented at \pm 45° with respect to the shearing section), measure specific linear deformations: TER1 measures $\epsilon = +45^\circ$, compression and TER2 measures $\epsilon = -45^\circ$, traction (fig. 2).

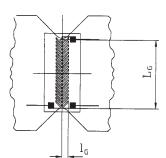


Fig. 2. Strain gauge rosette used for the measurement of losipescu specimen response to shearing.

The transducer signal is taken and amplified by a N2324 strain gauge bridge integrated into a data acquisition system (fig. 3).



Fig. 3. Acquisition system with integrated N2324 bridge

The supply voltage of the Wheatstone bridge is 1V so local heating of the material specimens (thermally insulated) and essential modification of the mechanical characteristics do not occur. When calibrating the strain gauge bridge, the transducer features and two supply voltages were taken into consideration: 4V for

$$\varepsilon_{cal,4V} = \frac{k_{punte}}{k_{TER}} \cdot 10000 \; (\mu m/m) \; , \text{ where } k_{bridge} = 2$$

and 1V for

$$\varepsilon_{cal,1V} = \frac{\varepsilon_{cal,4V}}{4} (\mu m/m)$$

C.

The actual deformation results from the specific deformation multiplied by 4 and by the cross sensitivity

$$\varepsilon_{1,2} = \frac{1 - v_0 K_t}{1 + K_t} \varepsilon_{1,2citit}$$

where v_0 is *Poisson ratio* of the material the manufacturer measured brand factor on (k_{TER}) : $v_0 = 0.285$. It is calculated:

$$\frac{1 - v_0 K_t}{1 + K_t} = \frac{1 - 0.285 \cdot 0.013}{1 + 0.013} = 0.98351$$

When calibrating the strain gauge bridge, we took into account the above mentioned data and we evaluated $\epsilon_{\text{bridge.calibration}}$ for an indication of the measurement instrument (millivoltmeter):

$$\varepsilon_{cal.punte} = 10000 \cdot \frac{3}{2.11} \cdot \frac{1}{4} = 2369,668 \mu m/m$$

To make sense of the obtained dimensionless values, the following relation is used:

$$1\mu m/m = 10^{-6} m/m = 10^{-6} = 1\mu\epsilon$$

The bridge strain gauge indication on the 10000 scale με is equivalent to the voltage measured in millivolts (maximum voltage is 10V = 10.000 mV).

The measurements were performed at a considered ambient temperature of 20°C.

The Wheatstone bridge was supplied with a voltage of 1V and was calibrated with a signal determined by the following relationship:

$$\varepsilon_{cal} = 10000 \frac{k_{punte}}{k_{TER}} \cdot \frac{U_P}{4(V)} =$$

$$= 10000 \cdot \frac{2}{2 \cdot 4} (\mu m/m) = 2500 (\mu m/m)$$

The strain gauge bridge indicated the specific shearing strain:

$$\gamma_{1,2} = \varepsilon_1 - \varepsilon_2$$

The bridge was read with a digital voltmeter.

Specimen strain was conducted so that not to reach the breaking point and the strain rate (the strain increase was less than 3N/s) was applied to provide compensation of the creep and allow a precise control of the machine (machine stop for deformation measurement).

Figure 4 shows the shape, dimensions and theoretical strain of the Iosipescu specimen.

To ensure the precision of strain gauge measurements, the dimensions of the shear specimens and the execution details were compliant to ASTM D 5379-93 recommendations.

The strain applied to the specimen is shearing by forces applied asymmetrically to the axis of the slots (vertical axis) using a suitable device.

Transversal shearing (in the specimen plane), denoted G_{12} is determined as the slope of curve τ_{12} - γ_{12} where:
- τ_{12} is the tangential tension in the shear section:

$$\tau_{12} = \frac{P}{wh}$$

 $\tau_{12} = \frac{P}{wh}$ - γ_{12} is specific sliding, which is expressed as the difference between the two specific elongations measured by the shear transducer rosette:

$$\gamma_{xy} = \varepsilon_{+45^{\circ}} - \varepsilon_{-45^{\circ}}$$

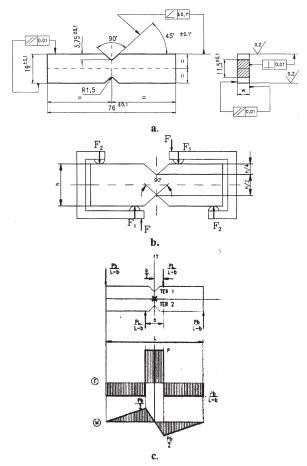


Fig. 4. The shape, size and strain of the Iosipescu specimen: a. Shear specimen dimensions and directions for execution; b. How to strain the specimen; c. Specimen strain and force diagrams

In the sense of norm ASTM D5379-93, the process is capable of providing accurate results for specimens having the shear plane oriented after a main trihedral plain.

Materials and specimen realisation

Based on relevant literature [18-22] 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. Similar to other polymers, the mechanical properties vary as the temperature changes. This material tends to creep. It is not suitable for operation under multiple dynamic loads, this being the reason for using reinforcements made of textiles or metal fibers [23, 24].
The Poly (methyl methacrylate) is very resistant to direct

sunshine exposure, the strength characteristics varying very little under UV radiation or oxygen/ozone.

In tables 1 to 3 are presented the properties of Poly (methyl methacrylate) and silicon materials involved in the research [23-27].

In addition to these polymers, for the doping of poly (metyl methacrylate), denoted PMMA, maleic anhydride (denoted MA) was used in a ratio of 1:3 (Sigma-Aldrich Chemical Co., Inc., Milwaukee, WI). In this case, 3-5mm thick PMMA and PMMA+MA plates were cast separately (1:3). These were adjusted by rotative grinding on abrasive surfaces until they became suitable as layered specimens (table 4). The two plates were bonded with CT-1 silicone

Table 1
TYPICAL PHYSICAL PROPERTIES OF POLY (METHYL
METHACRYLATE)

METTE CRIENTE)				
Value				
1.15 - 1.19				
0.3 - 2.0				
0.30 - 0.33				
0,003 - 0.0065				
0.9 – 27.0				
2.8 - 4.0				
3 - 4				
17.7 - 60.0				

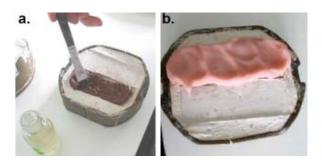


Fig. 5. Casting and specimen extraction

adhesive. Polyethylene fibre or copper mesh reinforcement was or was not placed between these plates. The reinforcement was immersed in silicone adhesive for bonding.

The test specimens were moulded from acrylic polymer in silicone rubber tanks. After hardening they were removed (fig. 5) and mechanically processed to obtain the standard shapes and dimensions of the Iosipescu tests.

Firstly, the study considered the optimization of aluminium mounting plates on the ends with silicone resin. This approach was tested using the shear test (fig. 6).

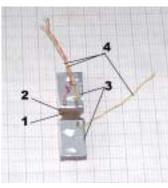




Fig. 6. Specimens and shear testing device: a - test specimens; b - the device; 1 shear rosette; 2 - specimen; 3 - aluminium fittings; 4 - cable connection to the strain gauge bridge

After installing the aluminium plates on the ends, the specimen shear rosette was fixed (electroresistive transducer TER).

In addition to the shear test meant to determine the mechanical characteristics involved in the structural-functional optimisation of matrix polymers used for removable prostheses, the losipescu traction method was carried out using a Textenser device (fig. 7).

Table 2
MECHANICAL CHARACTERISTICS OF POLYMETHYL METHACRYLATE

Mechanical characteristic	Value
Hardness, Rockwell M	63 - 97
Tensile Strength, Ultimate (MPa)	47 - 79
Elongation at Break (%)	1 - 30
Tensile Modulus (GPa)	2.2 - 3.8
Flexural Modulus (GPa)	3 - 3.5
Izod Impact, Notched (kJ/m²)	1.2 - 20.0
Izod Impact, Unnotched (kJ/m²)	11
Tensile Creep Modulus, 1 h (MPa)	1800 - 2700
Tensile Creep Modulus, 1000 h (MPa)	1200 - 1800

Table 3
THE CHARACTERISTICS OF SILICONE MATERIALS USED FOR SPECIMEN
MANUFACTURING

Characteristic of		the silicone material			
		Silicone rubber CMV-	Silicone adhesive		
		40	CT-1		
Hardness (OSHA	()	40±5	30±5		
Tensile strength (kgf/cm ²)		minimum 50	-		
Tensile strength (daN/cm ²)		-	14.50		
Elongation at break (%)		minimum 350	Minimum 300		
Detachment force (kgf / cm	Aluminium alloy foil	-	1.60		
(Kgf/cm ²) as against	Epoxy primer foil	-	1.50		
Work temperature range (°C)		-50 +200	-		
Dielectric constant, 50 Hz		-	2.82		
Dielectric strength (KW/mm)		-	20.0		



Fig. 7. The Textenser device used for tensile tests

Results and discussions

Two sets of test specimens were made for shear and tensile tests using poly (methyl methacrylate), denoted PMMA, with polyethylene fibre and copper wire mesh insertions.

In order to study the behaviour of the adhesive binding the plates and the reinforcement system, respectively the aluminium plates from the clamping jaw ends of the test specimens, the *tensile test* and the *shear test* were used

This study was needed to determine the dimensions of the surface the clamp jaws are applied on, the practical aspect that influences the accuracy of the final results, shown for five specimens with different compositions in tables 4 and 5.

Table 4 shows the structural composition and mechanical properties of specimens obtained using the tensile test, used for comparative study of the specimens' behaviour.

The notations used in table 4 are: d - width of the specimen between the V-shaped notches (middle), g - specimen thickness, very important for tensile behaviour,

Specimen		cimen	Dimensional characteristics		F _{max}	max	max	Tensile
	comp	position	d	g	N		real	behaviour
	Basic material	Reinforcement layout	mm	mm		MPa	MPa	Tear
0 reference	PMMA	without	18.8	2.90	1750	32.10	39.44	transverse under the jaw clamp
1	PMMA	longitudinally disposed polyethylene	21	1.45	1750	57.47	1	longitudinal offset
2	PMMA	longitudinally disposed mesh with d width	21	1.70	1125	31.51	82.72	transverse under the jaw clamp
3	PMMA// PMMA+AM	excepted	18.8	3.20	1300	21.60	26.55	transverse under the jaw clamp
4	PMMA// PMMA+AM	longitudinally disposed polyethylene	19.9	1.50	800	26.80	66.66	transverse under the jaw clamp
5	PMMA// PMMA+AM	longitudinally disposed mesh with d width	18.4	3.20	1650	28.02	3	transverse under the jaw clamp + cracks in the study area

Table 4 THE RESULTS OF SPECIMEN TRACTION

Specimen	Bonded area	Breaking strength	max (convențional)
	mm ²	N	MPa
0	$18.814 - (/4)(3,5)^2 = 253,584$	1750	5,083
1	$2114 - (/4)(3,5)^2 = 284384$	1750	5.656
2	$2114 - (/4)(3,5)^2 = 284.384$	1125	3,636
3	$18.814 - (4)(3,5)^2 = 253584$	1300	5.126
4	19.920-(/4)(3,5) ² =388,384	800	2.049
5	$18.415 - (/4)(3,5)^2 = 266,384$	1650	6.194

Table 5 PARAMETERS OF SPECIMENS BROKEN THROUGH SHEAR

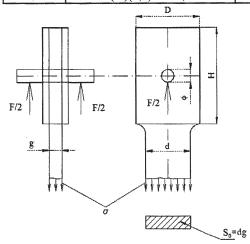


Fig. 8. The denotations used in the calculus of clamping head dimensions in relation to the shearing resistance of the adhesive used for the fixing of the aluminium plates for jaw clamping

 F_{max} - maximum force causing the specimen rupture, σ_{max} - maximum normal stress ($\sigma_{max}=F_{max}/S_0$), where $S_0=d$. g is the section where the breakage occurred (or the initial specimen section study area) σ_{max} real = actual maximum normal stress calculated for the section in which the actual breaking occurred. Here the tensile stress is often eccentric due to asymmetric structure and structure load distribution.

The values obtained for the maximum normal stress, σ_{max} vary between 22 and 60 MPa, depending on dimensional and structural composition of the specimens.

The data in table 4 confirm the role of stratification and inserts in relation to the tensile mechanical strength, dimensional and structural composition of specimens correlated with F_{max} and σ_{max} .

Table 5 shows the shear behaviour of the five specimens, shown in table 4.

Table 5 shows a tangential shear tension variation of the adhesive between 2 MPa and 6.2 MPa with an average of 4.624 MPa. The jaw clamp dimensions were established by a calculation based on the tensile strength of the specimen σ_{max} where the resulting maximum force is:

$$F_{\text{max}} = S_{\text{o}} = S_{\text{max}}$$
 where $S_{\text{o}} = D$.g

The maximum tangential tension:

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

wherein B and H are dimensions of the clamping heads and ϕ is the diameter of the holes in the grip head of the test specimen for the passage of the fastening bolt as shown in figure 8. The rated value of H_{min} is:

$$H_{\min} = \frac{\sigma_{\max}}{\tau_{\max}} \cdot \frac{dg}{2B} + \frac{\pi \phi^2}{4B}$$

 $H_{\min} = \frac{\sigma_{\max}}{\tau_{\max}} \cdot \frac{dg}{2B} + \frac{\pi \phi^2}{4B}$ If the tensile test is represented by obtained values of σ_{max} , the shear test is well represented by τ_{max} . These two are very important features used in correlation with the structural-functional characteristics of removable prosthetics.

Conclusions

This paper is the first note of a series that present the experimental protocol of optimization of structural and constructive characteristics of acrylic polymer specimen with insertions using the Iosipescu test. Tensile and shear tests allowed the determination of specimen consolidation with aluminium plates of clamping jaw heads. The experiment used silicone adhesives for simple removable acrylic polymer prosthetics or layered matrix systems.

Based on obtained experimental data and theoretical assessments, the following conclusions may be drawn:

-to assess the behaviour of the adhesive used for aluminium plates needed to fix the specimen ends, based on a theoretical protocol the two types of Isoipescu tests were selected for removable prostheses in polymeric matrices:

-the shape, size, stress of the Iosipescu specimen and sizing of the bridge were evaluated based on the same theoretical protocol;

-the study used two sets of specimens for shearing and traction tests using Poly(methyl methacrylate), denoted as PMMA. PMMA has polyethylene fibre and copper wire mesh inserts. These were individually assembled using CT-1 adhesive that was used to fix aluminium plates on the clamping jaw heads;

-the values obtained for the normal maximum traction stress, σ_{max} , ranged between 22 and 60 MPa, and the tangential shearing stress, τ_{max} , of the adhesive ranged between 2 and 6.2 MPa, with an average value of 4.624 MPa, both depending on the dimensional characteristics and structural composition of the specimens, thus confirming the role of stratification and inserts in the mechanical strength of the specimens;

- the tensile test, represented by the values of σ_{max} and shearing test represented by the values of τ_{max} was necessary to determine the dimensions of the surface the clamping jaws are applied on, a practical aspect influencing the accuracy of the final results.

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