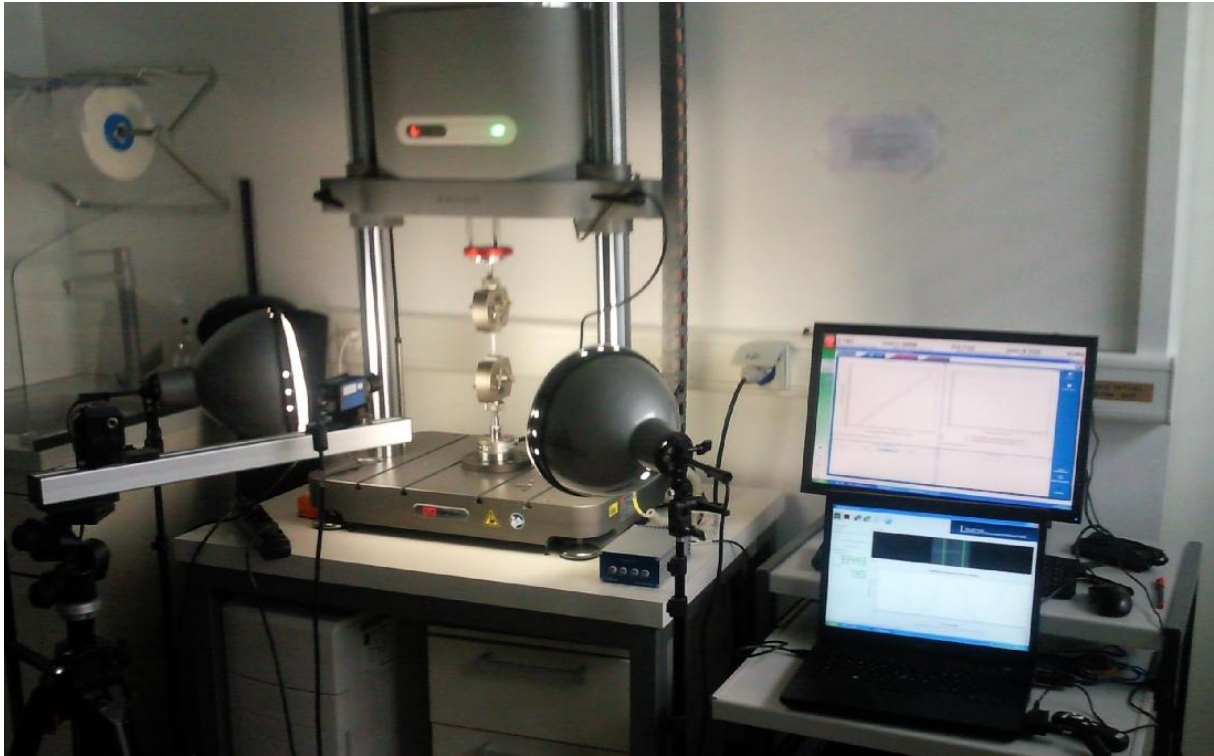


Characterization of viscoelastic properties Of PDMS silicon rubbers



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Table of Contents

Abstract 2

Introduction 2

Materials and Methods 4

 Mould assembly 4

 Samples preparation 5

 Tensile test 5

 Compression test 6

 Data analysis 7

Results and Discussion 7

 Samples preparation 8

 Tensile test 8

 Compression test 16

Conclusion 17

Acknowledgements 18

References 18

ABSTRACT

This work focuses on the viscous dissipation properties of polydimethylsiloxane (PDMS) relevant for encapsulation chambers for soft tissues and high deformable biomaterials mechanical tests applications. In view of the lack of dissipation consideration in biomaterials, and observing the increasing demand of such encapsulation chambers, we analyzed the dissipation of the commonly used PDMS (Sylgard 184 from Dow Corning). We focused on the effect of the thinner concentration and the polymerization temperature. We conclude that the PDMS dissipate less energy at a ratio of thinner of 1:5 and if polymerized at 37°C in tensile test. But in the chambers, in compression test, this material shows less energy dissipation at thinner ratio of 1:10 if polymerized at room temperature (20-25°C). Those results are indicatives because of the low number of tested samples and have to be verified by testing a large library of samples.

INTRODUCTION

Elastomers, one of the major classes of polymers are widely used in different applications of our life. They are used in MEMS (microelectromechanical systems) where they act as embedding or encapsulation structures of electronic components and microfluidics in chips. Elastomers are also used in the shoes industry for soles fabrication, in the car industry for tires production, in medicine for the fabrication of different types of devices, in tissue engineering for scaffolds production and many others applications. For all those applications, the mechanical properties such as young modulus, stiffness, Viscosity, hardness, tensile strength and thermodynamics properties were the main parameters focussed on. But as new applications for elastomers appear, some others viscoelastic considerations have to be taken into account. In fact in life sciences it is very difficult to do mechanical tests on soft tissues or biomaterial like hydrogels that are very difficult to handle. So there is a need of encapsulation devices to test this kind of soft material. Among the different elastomers used, the polydimethylsiloxane (PDMS), a viscoelastic elastomer, is the one which is currently used for such application as material.

The viscoelasticity is a consequence of the entropy production a given material and for a material to be considered as viscoelastic, it should present some phenomena such as the creep, the stress relaxation, the strain rate effect of the phase lag during tests.

For viscoelastic materials, the frictions in the material under cyclic mechanical tests give a mechanical hysteresis. Then using the linear theory the, viscous dissipation power of the material can be calculated with as follow:

$$\dot{Q}_{loss} = E^* \varepsilon 2 \pi \sin(\delta) f$$

Where E^* is the norm of the complex dynamic modulus, f the frequency, ε the strain, and δ the phase lag. Knowing the cycle time, the energy dissipated can then be found, because the power is the energy per unit of time. The energy dissipated can also be directly assessed by calculating the area of the hysteresis between the ascendant and the

descendant curves of the stress-strain plot obtain during test. This area can be obtained via the integral of the subtraction between those two curves.

For hydrogels in unconfined compression and shear the viscous dissipation power have been estimated in a range from 1 to 250 [$\mu\text{W}/\text{ml}$] for small deformations ($\leq 5\%$) [7]. so, for high deformations, high values are expected.

In this project we is to produce PDMS with the less viscous dissipation energy for the application of thin wall membrane uses as biocompatible chambers to support soft and high deformable sample biomaterials in large deformations mechanical compression tests. To achieve our aim we will play with two parameters when preparing the material such as the polymerization temperature or the crosslinking ratio.

MATERIALS AND METHODS

Moulds Assembly

Two types of moulds have been fabricated by my supervisor for this purpose.

For the shouldered test bars for the tensile test, the moulds are composed of three pieces in stainless steel: two outer pieces and the inner one with the chamber that give the shape to the sample. The three pieces were assembled and screwed together to give the functional mould as shown in the figure1 A.

For the cylindrical chambers for the compression test, The molds is composed of two outer pieces in a bi-component polyurethane (PU) resin (Neukadur multicast 20, Altrap, Germany) and the inner piece composed of two stainless steel parts assembled by a screw [7]. The assembly of the mould was done by lodging the inner piece in the outer ones and the assembled moulds are held together by a pair of pliers as shown in figure1B.



Figure1. A: Mould assembly process for the production of the shouldered test bar. B: Mould assembly process for the production of the encapsulation chamber production.

Samples preparation

The sylgard 184 base and hardener (Sylgard 184, Dow Corning Corporation, USA) were weighted at different weight ratios (5:1; 10:1; 15:1 and 20:1). The compounds were mixed in a beaker for several minutes with a spatula. The preparations were degassed in the desiccators under the vacuum until there were no more bubbles in the bulk. The moulds were incubated in the oven for around 30minutes prior to casting (during the degassing process). The compounds were then poured into the moulds using injection casting and avoiding formation of air bubbles when transferring it in the syringe. The moulds with samples are incubated at different temperatures (room temperature; 37°C; 40°C and 70°C) in the oven or at room temperature for several hours or days. The excess of PDMS on the side was used to control the completeness of the process. The samples were then taken out of the moulds, the excess of PDMS on the sides removed using a scalpel and the samples stored in 50ml tubes.

Tensile Test

The tensile test was performed using the INSTRON machine (Electropulse E3000, Instron, USA) and a camera (Vic2D, Limesh GmbH, Germany). The test consists on maintaining one end of the sample fixe while pulling on the other on. And the machine equipped of sensors determines the stress corresponding to the applied strain and compute the corresponding hysteresis

The set up for the test were assembled. The software for the control of the INSTRON machine and the camera were switched on and those equipments are calibrated via their respective softwares. The Samples were prepared for test by drawing on some arrows that delimit the tested region. The lines delimiting the loading region were drawn at 15mm from each end and the lines delimiting the test zone were drawn at 12,5 mm of the previews ones as shown in figure2C. The samples were then loaded on the INSTRON machine according to the delimited zones as in the figure2A. The upper and lower positions and digital positions of the moving part (upper) of the machine were set to calibrate the zero displacement and the zero force with the height of the undeformed sample. The oscillatory test is set to a sine shape and the amplitude of the strain for the test was set to 14% (corresponding to an elongation of 26,4 mm) in the test zone. The camera was used to have a feedback when adjusting the strain amplitude in the test zone because the shouldered test bar doesn't have a regular shape, the strain is not equally distributed in the sample so the camera with markers that recognize the two lines delimiting the test zone and thus gives on the software controlling the camera, the value of the strain that is really applied on this zone as shown in figure2B. The cycle time was set to 5S the initial phase is set to 270° because the sine is at it minimum at this value, thus the sample remains in tension during the whole test and it will never bend. Each sample was then tested with several cycles (from 20 for the low frequencies, to 200 for high frequencies) of the oscillatory sine shaped strain at four different frequencies (0.2Hz; 1Hz; 5Hz and 10Hz). Data were recorded and saved for the subsequent analysis.

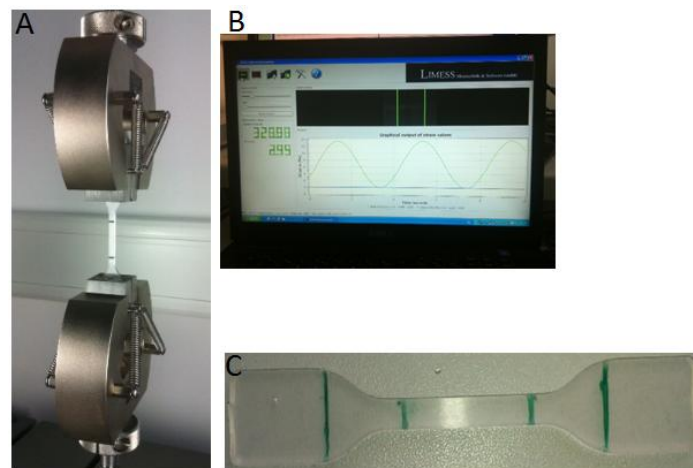


Figure2. A: the shouldered test bar on loaded on the machine during tensile test. B: the returned feedback from the LIMESS camera for the control of the strain amplitude during tests. C: The shouldered test bar with the test labels (the two outer marks are separated by 45mm while the two inner ones are separated by 20mm)

Compression Test

The compression test was performed on the same machine (Electropulse E3000, Instron, USA) used for the tensile test. The test consisted on maintaining one end of the closed chamber filled with water fixe, and compriming cyclically on the other end as shown in figure (fig). Then the machine equipped with sensors senses the responding stress force to the applied strain, sends it to the software that compute the corresponding hysteresis.

The different apparatus of the test set up were switched on and the INSTRON machine calibrated via its software. The samples were closed at hermetically at one end using a special circular piece in steel, filled with water and closed on the other end with another circular piece in steel with a small hole in the center. The two circular pieces at the ends permit also to fermly grip the tested chambers on the instron machine when loading them. The samples were then loaded on the machine as shown in the figure3. The syringe filled with water was connected to the inlet of the machine, an additional amount of water added in the chamber and the syringe kept there during the whole testing process in order to maintain the walls of the chambers in tension during the test. The water was used to fill the chambers because it is known to no dissipate energy and thus making us be sure that the dissipations provided by the test are from the material used for the chambers. The zeros displacement and force were set, as well as the initial phase for the test set at 90° because we want to compress the sample so we start with the higher value of the sine function which is equal to 1 at 90° . The amplitude of the oscillatory strain was set to 15%. Here the camera is not used because the test zone was regularly shaped and the walls maintained in radial tension owed to the water filled in. Thus the feedback when setting the strain amplitude was not needed because it was evidence that the set amplitude was the one that was really applied in the test zone. The cycle time was set to 0,02s. The samples were then tested with an oscillatory compressive strain at three different frequencies four variable number of

cycles (from 30 for low frequencies, up to 100 for high frequencies). Data are finally registered and saved for the subsequent analysis

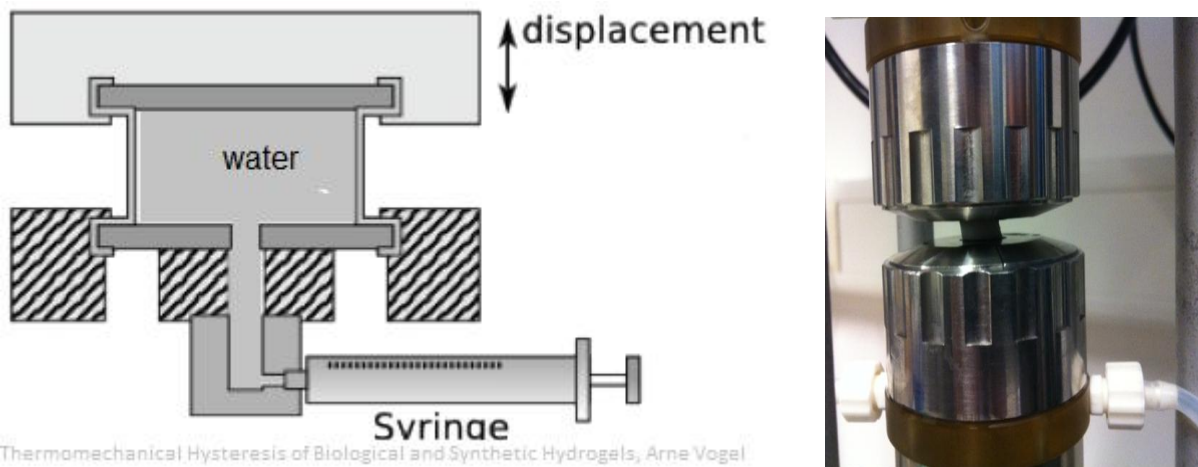


Figure3. The set up for the compression test. Left: schematic description of the set up with the chamber filled with water, the injection system the fixed part of the machine and the moving (testing) one. Right: The chamber (central part) mounted on the machine during compression test.



Figure4. The top view (left) and the side view (right) of the chamber for the compression test.

Data analysis

For the analysis, the work.m file was used. This file contains the functions that compute and plot the hysteresis (stress-strain curves in ascending and descending phases of the sine) and the values of the dissipated energy (against the cycles numbers) in each cycle.

Data acquired during the tests were imported in matlab then the different functions of the work.m file were run successively as following: `` work (data); `` after that ``w = work (data) `` , ``methods (w) `` , then ``w.hysteresis`` and ``w.plot``. Then for each sample and for each frequency, the average power loss over all the cycles was estimated, the standard deviation in each condition computed using the following formula:

$$\sigma = \sqrt{\frac{\sum_{i=1}^n (x_i - X)^2}{n-1}}$$

Where xi is the mean power loss for one sample with a given ratio at one test condition, X the average of the mean powers of all the samples with a given the same ratio at the same test condition and n the total number of samples with this ratio and tested at the same

conditions. The volumes of the samples (volume between the two outer marks for the shouldered bar and volume of the ventral part of the chamber) were then computed and the volumic dissipations were finally calculated by dividing the energy dissipated by the volume. Histograms were then plotted using the excel software to show the effect of the change of composition ratios or the polymerization temperature on the power dissipation on our material.

RESULTS AND DISCUSSION

Samples preparation

For the for ratios (1:5; 1:10; 1:15 and 1:20) selected for this work for the different temperature of hardening (room temperature; 37°C and 70°C), 15 shouldered bars and 28 chambers were casted. But all the 15 shouldered bars were demolded successfully and only 7 chambers. This corresponds to a recovery percentage of 100% for shouldered bars and 25% for the encapsulation chambers. This difference could be explained by difference in the thickness of the two types of samples and also the materials used to manufacture their moulds. In fact the shouldered bars are 2mm thick and casted in mould made in steel while the chambers walls are only 0.1mm thick and casted in mould made in polyurethane. It seems that polyurethane which also an elastomer like our PDMS reacts more with the injected material. Also any chamber was successfully demolded at polymerization temperatures higher neither than 40°C nor for ratios lower than 1:10 (1:15 and 1:20). It seems that for those ratios the hardener is so small that the crosslinking is low and thus making the material difficult to handle. This is why it breaks when demolding. The lower number of casted samples is due to the fact that only two moulds for shouldered bars and six for encapsulation chambers were available. And the material was polymerized for an average of three days each batch in order to be sufficiently stiff to be handled.

Tensile test

For the tensile test, after the data treatment on matlab, the stress (Force) – strain (displacement) curves of the different samples were plotted. Here are some of them (figure5)

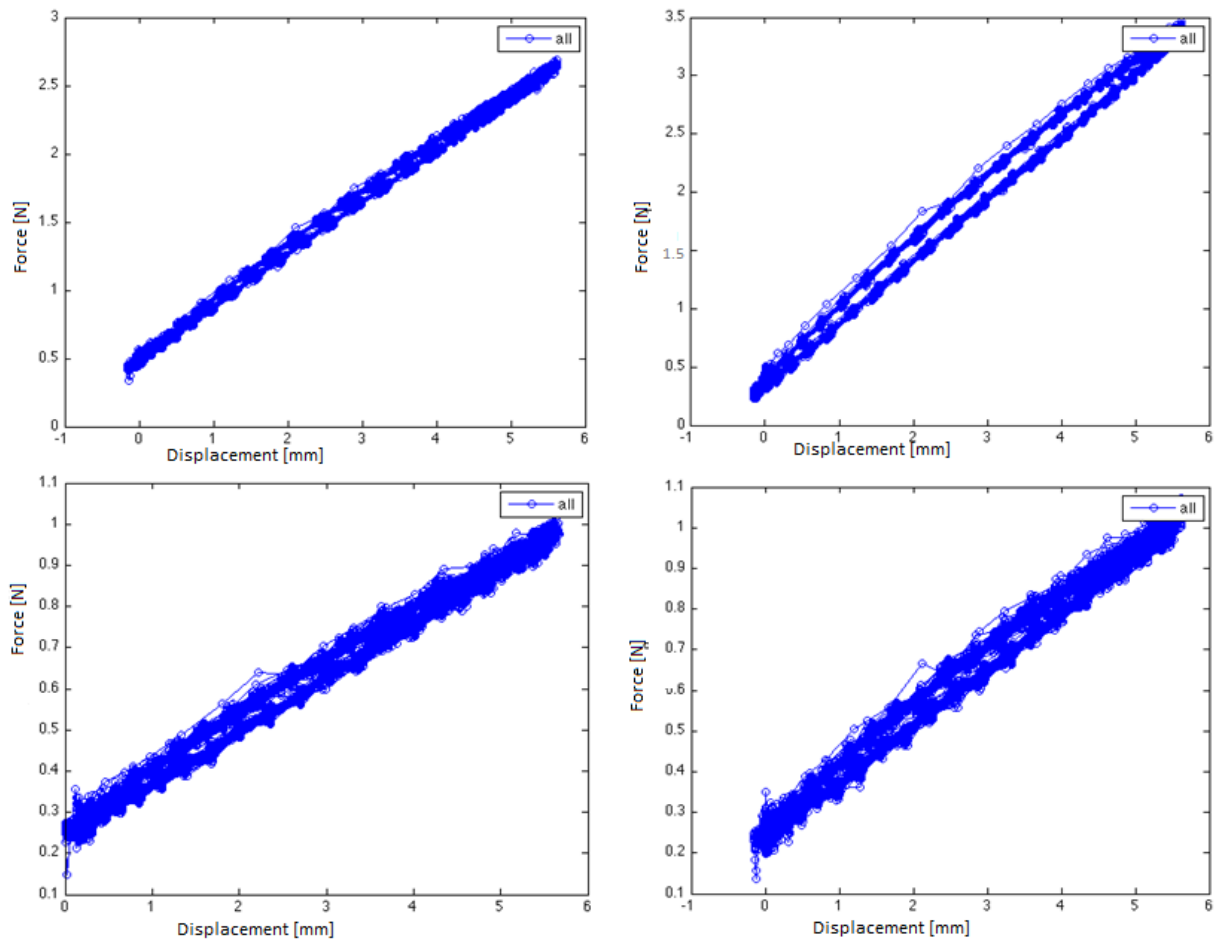


Figure5. typical stress – strain cuves for the four ratios tested : 1:5 (uppe left), 1:10 (upper right), 1:15 (down left) and 1:20 (down right). For the prsented in this figure, the polymerisation temperature was 37°C and the test frequency 1Hz

From figure5 it is easy to remark that the stress- strain curves in the ascending(upper blue curves in each of the four squares) and in the descending(lower blue curves in each of the four squares) sine don't coincide. So there is a kind of gap(hysteresis) between the two curves . the area of this hysteresis can be calculated by subtracting t the integral of the ascendant cuve and the integral of the descending cuve. The hyseresis area calculated here corresponds to the energy dissipated. Here in figure5 in each of the four plots, at least 100 hundred cycles are plotted, thus at least 100 hysteresis for each sample are plotted. Visually, the sample at 1:10 seems to have the highest area while the one at 1:5 shows the lowest area and 1:15 and 1:20 seem to be in between.

For each test, the value of the hysteresis or the dissipated energy (area of the hysteresis) for each test cycle were computed. The energies dissipated were then plotted against their corresponding cycles. Here are some of them (Figure6). The curves presented in figure6 were obtained from those on figure5

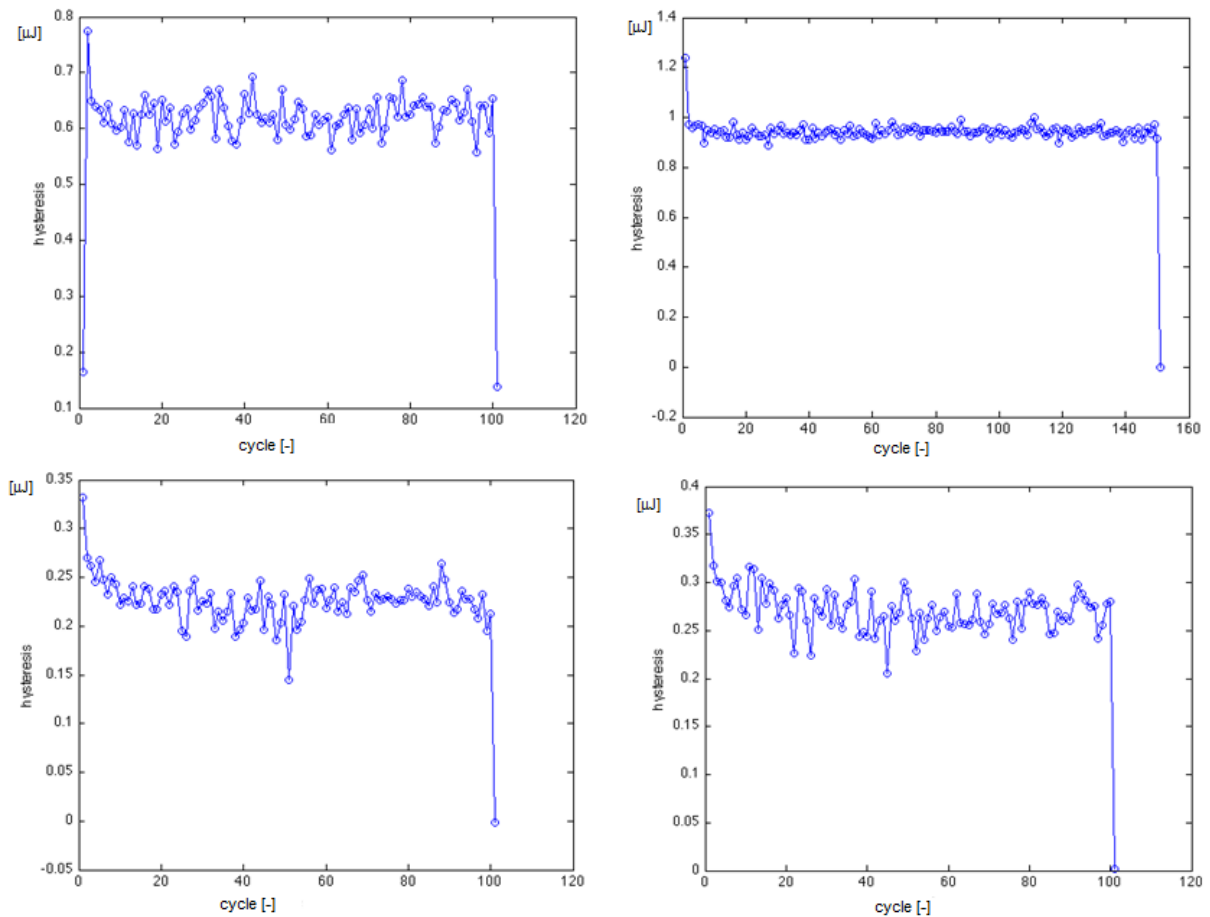


Figure 6. Dissipated energy against cycles number plots obtained from figure 5. 1:5 (upper left), 1:10 (upper right), 1:15 (down left) and 1:20 (down right). The polymerization temperature in this case were 37°C and the test frequency 1Hz. The dissipation (hysteresis here) is in μJ

From figure 6, the sample with a 1:10 ratio seems to be as visually observed in figure 5 the one with the highest dissipation. But instead of 1:5, it is the one at 1:15 which seems to dissipate less, followed by 1:20 and 1:5. Also as it can be observe in figure 5 the curves for 1:10 are close packed while the other ones fluctuate much. This is also observed on figure 6 with the 1:10 curve which is quasi-linear while the others fluctuate.

For each test frequency, the averages dissipations (over all the cycles) for the different samples were estimated on the curve and then reported on tables (tables: 1, 3, 5 and 7). Then average dissipations of all the samples with the same ratios, polymerized at the same temperatures and tested at the same frequencies were averaged and reported in the same tables (tables: 1, 3, 5 and 7) as well as the volume of the sample. The different dissipation from tables 1,3,5,7 were then divided by the volume of the samples which is the same to yield the volumic dissipations which were also averaged, their standard deviations calculated and reported in tables (tables: 2, 4, 6, 8)

Table1: Dissipation estimated form hysteresis curve for each sample and the volume of the sample for tensile test performed at 0,2Hz

| | Dissipation (J) 1:10_RT | Dissipation (J) 1:10_70 | Dissipation (J) 1:10_37 | Dissipation (J) 1:15_37 | Dissipation (J) 1:15_RT | Dissipation (J) 1:20_37 | Dissipation (J) 1:5_37 | volume(m ³) |
|--------------------|----------------------------|----------------------------|----------------------------|----------------------------|----------------------------|----------------------------|---------------------------|-------------------------|
| 0,2Hz | 5,65E-07 | 7,50E-07 | 5,90E-07 | 3,10E-07 | 1,50E-07 | 3,05E-07 | 1,85E-07 | 5,30E-07 |
| | 5,70E-07 | 7,45E-07 | 5,80E-07 | 3,20E-07 | | 3,00E-07 | 1,90E-07 | 5,30E-07 |
| | 5,80E-07 | 7,70E-07 | 5,95E-07 | 3,25E-07 | | 3,10E-07 | 1,80E-07 | 5,30E-07 |
| | 5,10E-07 | | 6,50E-07 | 5,00E-07 | | 1,80E-07 | 2,35E-07 | 5,30E-07 |
| | | | 6,45E-07 | 4,95E-07 | | | 2,45E-07 | 5,30E-07 |
| | | | 6,40E-07 | 5,15E-07 | | | 2,40E-07 | 5,30E-07 |
| | | | 6,00E-07 | | | | 2,25E-07 | 5,30E-07 |
| Average | 5,56E-07 | 7,55E-07 | 6,14E-07 | 4,11E-07 | 1,50E-07 | 2,74E-07 | 2,14E-07 | 5,30E-07 |
| Standard deviation | 3,14576E-08 | 1,32288E-08 | 2,94998E-08 | 1,01657E-07 | - | 6,26332E-08 | 2,82E-08 | - |

Table2: Volumic dissipations calculated from table1, their averages and standard deviations for each sample for tensile test performed at 0,2Hz

| | volumic dissipation(J/m ³) 1:10_RT | volumic dissipation(J/m ³) 1:10_70 | volumic dissipation(J/m ³) 1:10_37 | volumic dissipation(J/m ³) 1:15_37 | volumic dissipation(J/m ³) 1:15_RT | volumic dissipation(J/m ³) 1:20_37 | volumic dissipation(J/m ³) 1:5_37 |
|--------------------|---|---|---|---|---|---|--|
| 0,2Hz | 1,07E+00 | 1,42E+00 | 1,11E+00 | 5,85E-01 | 2,83E-01 | 5,75E-01 | 3,49E-01 |
| | 1,08E+00 | 1,41E+00 | 1,09E+00 | 6,04E-01 | | 5,66E-01 | 3,58E-01 |
| | 1,09E+00 | 1,45E+00 | 1,12E+00 | 6,13E-01 | | 5,85E-01 | 3,40E-01 |
| | 9,62E-01 | | 1,23E+00 | 9,43E-01 | | 3,40E-01 | 4,43E-01 |
| | | | 1,22E+00 | 9,34E-01 | | | 4,62E-01 |
| | | | 1,21E+00 | 9,72E-01 | | | 4,53E-01 |
| | | | 1,13E+00 | | | | 4,25E-01 |
| Average 0,2Hz | 1,05E+00 | 1,42E+00 | 1,16E+00 | 7,75E-01 | 2,83E-01 | 5,17E-01 | 4,04E-01 |
| Standard deviation | 0,059354044 | 0,024959918 | 0,055659997 | 0,191805855 | - | 0,118175833 | 0,053207483 |

Table3: Dissipation estimated form hysteresis curve for each sample and the volume of the sample for tensile test performed at 1Hz

| | Dissipation (J) 1:10_RT | Dissipation (J) 1:10_70 | Dissipation (J) 1:10_37 | Dissipation (J) 1:15_37 | Dissipation (J) 1:15_RT | Dissipation (J) 1:20_37 | Dissipation (J) 1:5_37 | volume(m ³) |
|--------------------|----------------------------|----------------------------|----------------------------|----------------------------|----------------------------|----------------------------|---------------------------|-------------------------|
| 1Hz | 8,30E-07 | 1,10E-06 | 8,40E-07 | 4,75E-07 | 2,30E-07 | 4,55E-07 | 3,15E-07 | 5,30E-07 |
| | 8,25E-07 | 1,11E-06 | 9,10E-07 | 4,65E-07 | | 4,40E-07 | 3,10E-07 | 5,30E-07 |
| | 8,20E-07 | 1,09E-06 | 9,05E-07 | 4,70E-07 | | 4,35E-07 | 3,05E-07 | 5,30E-07 |
| | 7,90E-07 | | 9,90E-07 | 7,80E-07 | | 2,70E-07 | 3,10E-07 | 5,30E-07 |
| | | | 9,80E-07 | 7,50E-07 | | | 3,10E-07 | 5,30E-07 |
| | | | 9,90E-07 | 7,75E-07 | | | 3,05E-07 | 5,30E-07 |
| | | | 9,80E-07 | | | | 3,20E-07 | 5,30E-07 |
| | | | | | | | 3,15E-07 | 5,30E-07 |
| | | | | | | | 3,70E-07 | 5,30E-07 |
| Average | 8,16E-07 | 1,10E-06 | 9,42E-07 | 6,19E-07 | 2,30E-07 | 4,00E-07 | 3,18E-07 | 5,30E-07 |
| Standard deviation | 1,79699E-08 | 0,00000001 | 5,81562E-08 | 1,6375E-07 | - | 8,70823E-08 | 2,01729E-08 | - |

Table4: Volumic dissipations calculated from table3, their averages and standard deviations for each sample for tensile test performed at 1Hz

| | volumic dissipation(J/m ³) 1:10_RT | volumic dissipation(J/m ³) 1:10_70 | volumic dissipation(J/m ³) 1:10_37 | volumic dissipation(J/m ³) 1:15_37 | volumic dissipation(J/m ³) 1:15_RT | volumic dissipation(J/m ³) 1:20_37 | volumic dissipation(J/m ³) 1:5_37 |
|--------------------|---|---|---|---|---|---|--|
| 1Hz | 1,57E+00 | 2,08E+00 | 1,58E+00 | 8,96E-01 | 4,34E-01 | 8,58E-01 | 5,94E-01 |
| | 1,56E+00 | 2,09E+00 | 1,72E+00 | 8,77E-01 | | 8,30E-01 | 5,85E-01 |
| | 1,55E+00 | 2,06E+00 | 1,71E+00 | 8,87E-01 | | 8,21E-01 | 5,75E-01 |
| | 1,49E+00 | | 1,87E+00 | 1,47E+00 | | 5,09E-01 | 5,85E-01 |
| | | | 1,85E+00 | 1,42E+00 | | | 5,85E-01 |
| | | | 1,87E+00 | 1,46E+00 | | | 5,75E-01 |
| | | | 1,85E+00 | | | | 6,04E-01 |
| | | | | | | | 5,94E-01 |
| | | | | | | | 6,98E-01 |
| Average 1Hz | 1,54E+00 | 2,08E+00 | 1,78E+00 | 1,17E+00 | 4,34E-01 | 7,55E-01 | 6,00E-01 |
| Standard deviation | 0,033905438 | 0,018867925 | 0,109728667 | 0,308962864 | - | 0,164306295 | 0,038062008 |

Table5: Dissipation estimated form hysteresis curve for each sample and the volume of the sample for tensile test performed at 5Hz

| | Dissipation (J) 1:10_RT | Dissipation (J) 1:10_70 | Dissipation (J) 1:10_37 | Dissipation (J) 1:15_37 | Dissipation (J) 1:15_RT | Dissipation (J) 1:20_37 | Dissipation (J) 1:5_37 | volume(m ³) |
|--------------------|----------------------------|----------------------------|----------------------------|----------------------------|----------------------------|----------------------------|---------------------------|-------------------------|
| 5Hz | 1,41E-06 | 1,41E-06 | 1,12E-06 | 6,45E-07 | 2,80E-07 | 5,75E-07 | 4,75E-07 | 5,30E-07 |
| | 1,10E-06 | 1,42E-06 | 1,18E-06 | 6,50E-07 | | 5,85E-07 | 4,55E-07 | 5,30E-07 |
| | 1,10E-06 | | 1,16E-06 | 6,40E-07 | | 5,80E-07 | 4,80E-07 | 5,30E-07 |
| | 1,09E-06 | | 1,23E-06 | 9,70E-07 | | 4,75E-07 | 5,10E-07 | 5,30E-07 |
| | 1,18E-06 | | 1,24E-06 | 9,60E-07 | | | 5,20E-07 | 5,30E-07 |
| | | | 1,23E-06 | 9,80E-07 | | | 6,20E-07 | 5,30E-07 |
| | | | 1,22E-06 | | | | | 5,30E-07 |
| Average | 1,18E-06 | 1,42E-06 | 1,20E-06 | 8,08E-07 | 2,80E-07 | 5,54E-07 | 5,10E-07 | 5,30E-07 |
| Standard deviation | 1,36473E-07 | 7,07107E-09 | 4,49868E-08 | 1,7815E-07 | - | 5,26585E-08 | 5,89067E-08 | - |

Table6: Volumic dissipations calculated from table5, their averages and standard deviations for each sample for tensile test performed at 5Hz

| | volumic dissipation(J/m ³) 1:10_RT | volumic dissipation(J/m ³) 1:10_70 | volumic dissipation(J/m ³) 1:10_37 | volumic dissipation(J/m ³) 1:15_37 | volumic dissipation(J/m ³) 1:15_RT | volumic dissipation(J/m ³) 1:20_37 | volumic dissipation(J/m ³) 1:5_37 |
|--------------------|---|---|---|---|---|---|--|
| 5Hz | 2,66E+00 | 2,65E+00 | 2,11E+00 | 1,22E+00 | 5,28E-01 | 1,08E+00 | 8,96E-01 |
| | 2,08E+00 | 2,67E+00 | 2,23E+00 | 1,23E+00 | | 1,10E+00 | 8,58E-01 |
| | 2,07E+00 | | 2,19E+00 | 1,21E+00 | | 1,09E+00 | 9,08E-01 |
| | 2,06E+00 | | 2,32E+00 | 1,83E+00 | | 8,96E-01 | 9,62E-01 |
| | 2,23E+00 | | 2,34E+00 | 1,81E+00 | | | 9,81E-01 |
| | | | 2,32E+00 | 1,85E+00 | | | 1,17E+00 |
| | | | 2,30E+00 | | | | |
| Average 5Hz | 2,22E+00 | 2,66E+00 | 2,26E+00 | 1,52E+00 | 5,28E-01 | 1,04E+00 | 9,62E-01 |
| Standard deviation | 0,257497058 | 0,013341637 | 0,084880699 | 0,336132486 | - | 0,099355643 | 0,111144728 |

Table7: Dissipation estimated form hysteresis curve for each sample and the volume of the sample for tensile test performed at 10Hz

| | Dissipation (J) 1:10_RT | Dissipation (J) 1:10_70 | Dissipation (J) 1:10_37 | Dissipation (J) 1:15_37 | Dissipation (J) 1:15_RT | Dissipation (J) 1:20_37 | Dissipation (J) 1:5_37 | volume(m ³) |
|------|----------------------------|----------------------------|----------------------------|----------------------------|----------------------------|----------------------------|---------------------------|-------------------------|
| 10Hz | 1,10E-06 | | 6,20E-07 | | 9,50E-07 | 6,00E-07 | 4,90E-07 | 5,30E-07 |

Table8: Volumic dissipations calculated from table7, their averages and standard deviations for each sample for tensile test performed at 10Hz

| | volumic dissipation(J/m ³) 1:10_RT | volumic dissipation(J/m ³) 1:10_70 | volumic dissipation(J/m ³) 1:10_37 | volumic dissipation(J/m ³) 1:15_37 | volumic dissipation(J/m ³) 1:15_RT | volumic dissipation(J/m ³) 1:20_37 | volumic dissipation(J/m ³) 1:5_37 |
|------|---|---|---|---|---|---|--|
| 10Hz | 2,08E+00 | | 1,17E+00 | | 1,79E+00 | 1,13E+00 | 9,25E-01 |

The tables 2, 4, 6, 8 were then used to plot the histograms (figure7) in order to show the effect of the different parameters manipulated.

To show the crosslinker effect histograms were drawn for the different test frequencies (figure 7) for samples prepared at the same temperature in order to avoid the interference of another parameter.

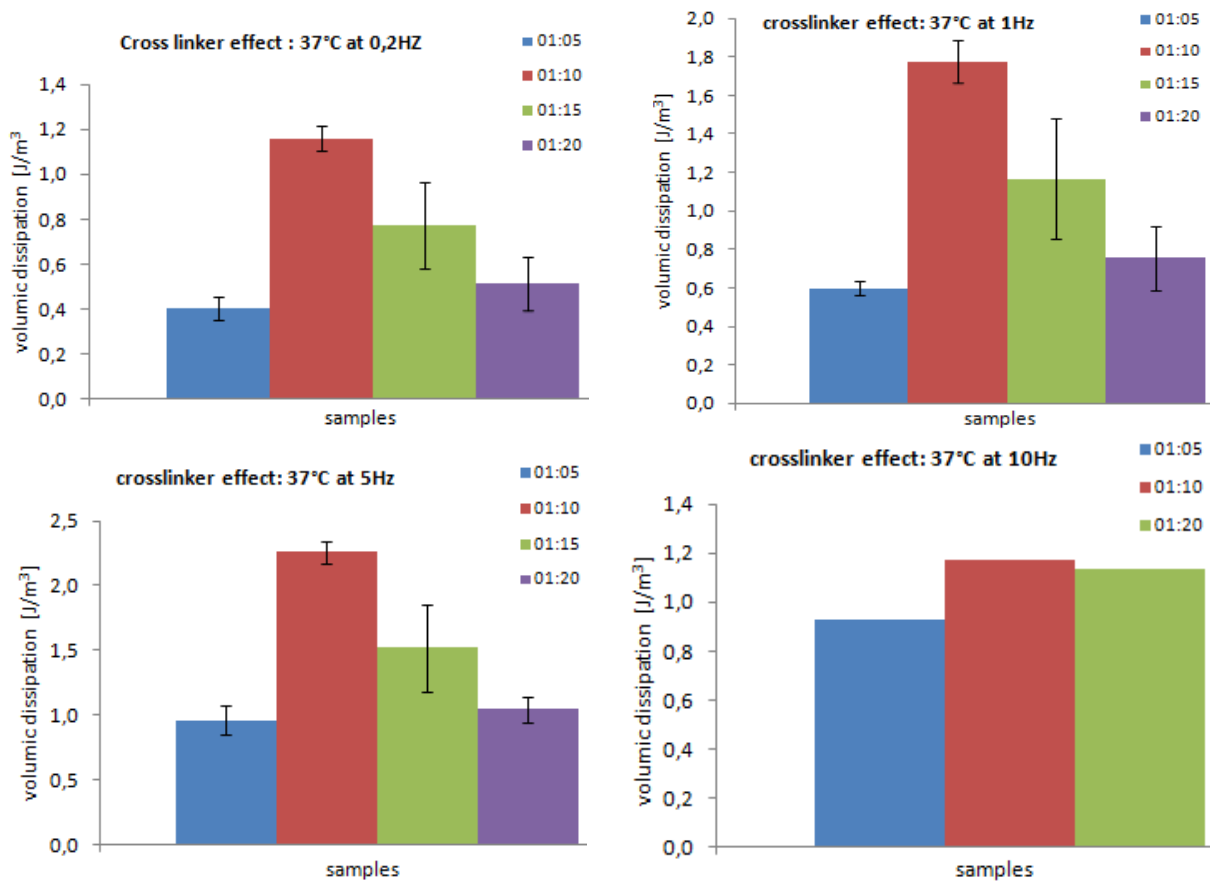


Figure7. Histograms showing the crosslinker effect on the dissipation of the material: All the samples presented here were polymerized at the same temperature (37°C).

As it is shown in those histograms for the test at 0,2; 1 and 5Hz the ratio of 1:10 seems to significantly dissipate more than all the other ratios. This is an unexpected result. Because an increase in dissipation with the decrease in hardener content in the material were expected. Thus the ratios 1:15, and 1:20 where there should be more entropy due to low crosslinking should dissipate more than the 1:10. The ratio 1:5 seems to be the one which dissipate less followed by the 1:20 and the 1:15. But when looking at the error bars, the dissipation difference between these three ratios seems to not be significant for the test performed at 0,2Hz and 1Hz. Because the error bars of 1:20 intersect with those of 1:5 and 1:15. however the ratio 1:5 seems to significantly dissipate less than the 1:15 at frequencies up to 5 Hz. For 5Hz, 1:20 seems to significantly dissipate less than 1:15 while remaining not significantly different to 1:5 as at 0,2Hz and 1Hz. Only three samples were tested at 10Hz because as it is shown in figure8 the shape of the hysteresis obtained at this frequency was not as expected

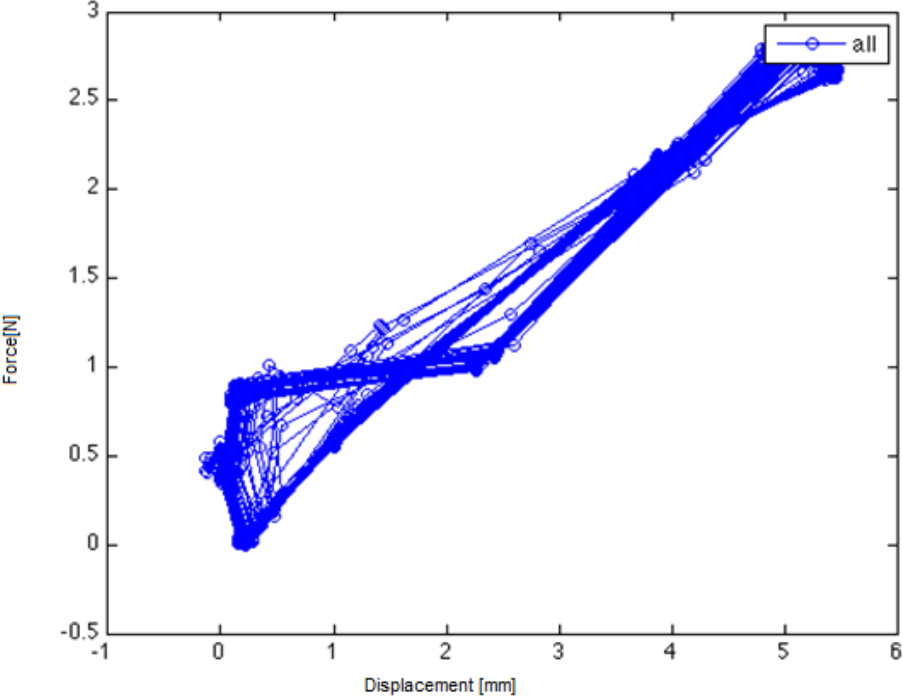


Figure8. Typical hysteresis obtained for test performed at 10Hz

To show the effect of the polymerization temperature on the dissipation of the material, histograms were plotted with the dissipations of the samples prepared at the same mixing ratio but at different polymerization temperatures for three frequencies

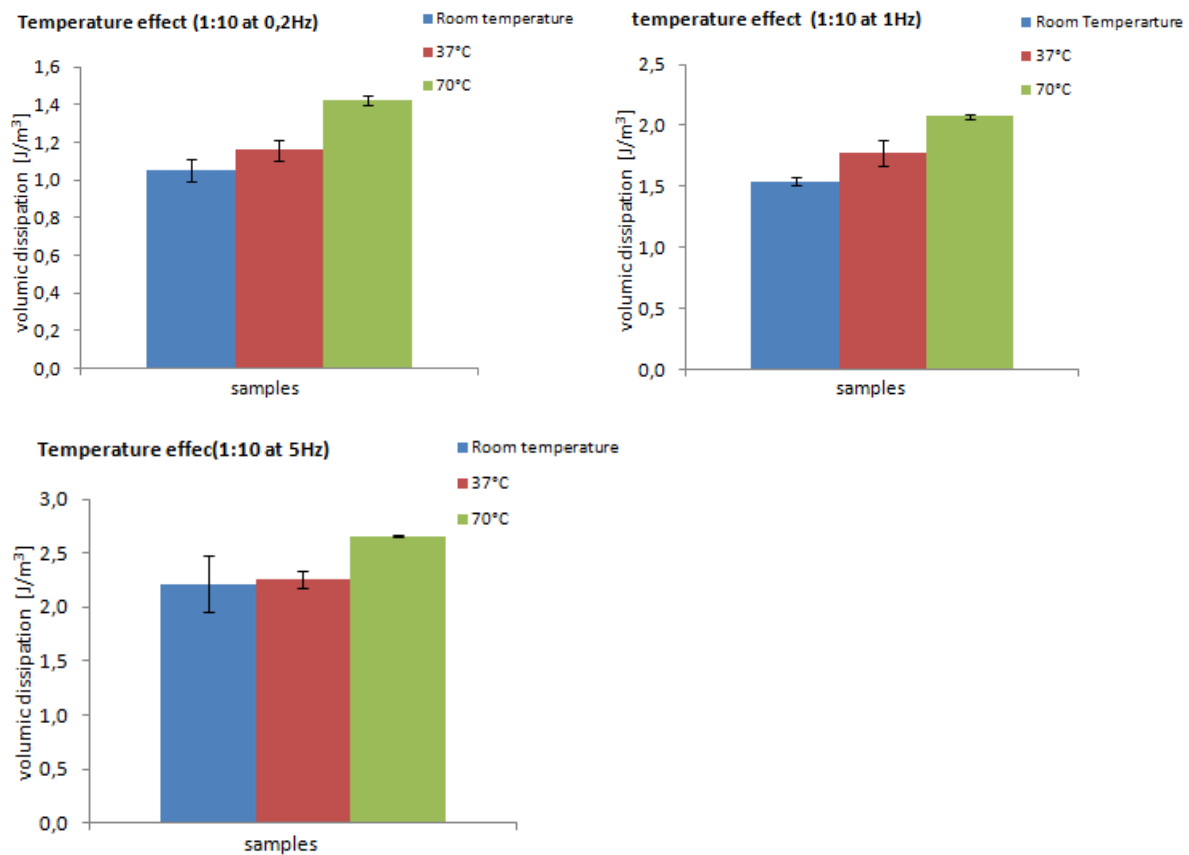


Figure 9. The effect of polymerization temperature on the dissipation of the material. All the samples presented here were polymerized at the same ratio, 1:10 but different temperatures of polymerization (Room temperature, 37°C and 70°C).

From the histograms above (figure 9), at all the tested frequencies, the dissipation at 70°C seems to be significantly higher than those at room temperature and 37°C but the dissipations at room temperature and 37°C seem to be not significantly different. It seems that a small increase in polymerization temperature doesn't have any effect on the dissipation (going from room temperature of around 25°C to 37°C). While after a certain threshold the increase in polymerization begins to increase the dissipation (from room temperature or 37°C to 70°C). This might be explained by the fact that the elevated temperature increases the entropy in the material. Thus the material polymerizing at high temperatures polymerize in a disordered state that remains even after polymerization and thus giving high dissipation values

By looking the crosslinker effect and the polymerization temperature effect histograms, it can be seen that the testing frequency influences the dissipation. It is clear on all those histograms that, the increase in the test frequencies increases the dissipation of the material. Thus the strain rate has an influence on the dissipation of the material. This might be explained by the fact that at low frequencies the material deformed has enough time to rearrange its molecules and thus there is less entropy when performing the following cycle.

While at high frequencies the time to rearrange the molecules in the material between two consecutive cycles is not enough and thus there is high entropy in the material in the next cycle and high dissipation.

Compression test

For compression test the encapsulation chambers were used. The stress – strain curves were plotted and had the same shape as those shown in figure5. Then hysteresis were also computed and plotted against their cycles numbers as in tensile test and those curves had also the same shapes as those presented in figure6. As with the tensile test data the average dissipations were estimated from the curves, the volume of the membrane of the tested zone of the encapsulation chambers calculated. Finally the volumic dissipations were calculated and reported in a table (table9). Here the averaged volumic dissipations and standard deviations were not computed because we performed only on test for each frequency and each sample. Only three samples were tested

Table9: Results obtained for compression tests.

| | 1:5_37°C | 01:10_37°C | 1:10_RT | Volume(m ³) | 1:5_37°C | 1:10_37°C | 1:10_Room temperature |
|-------|----------------|----------------|----------------|-------------------------|--|--|--|
| | Dissipation(J) | Dissipation(J) | Dissipation(J) | | volumic dissipation(J/m ³) | volumic dissipation(J/m ³) | volumic dissipation(J/m ³) |
| 0,5Hz | 1,80E-08 | 4,50E-08 | 1,50E-08 | 8,4823E-09 | 2,12E+00 | 5,31E+00 | 1,77E+00 |
| 1Hz | 2,90E-08 | 5,00E-08 | 2,00E-08 | 8,4823E-09 | 3,42E+00 | 5,89E+00 | 2,36E+00 |
| 5Hz | 3,10E-08 | 6,00E-08 | 2,80E-08 | 8,4823E-09 | 3,65E+00 | 7,07E+00 | 3,30E+00 |

From the table above (table9) one histogram was plotted to investigate the effect of the crosslinker and the temperature on the dissipation of the encapsulation chambers.

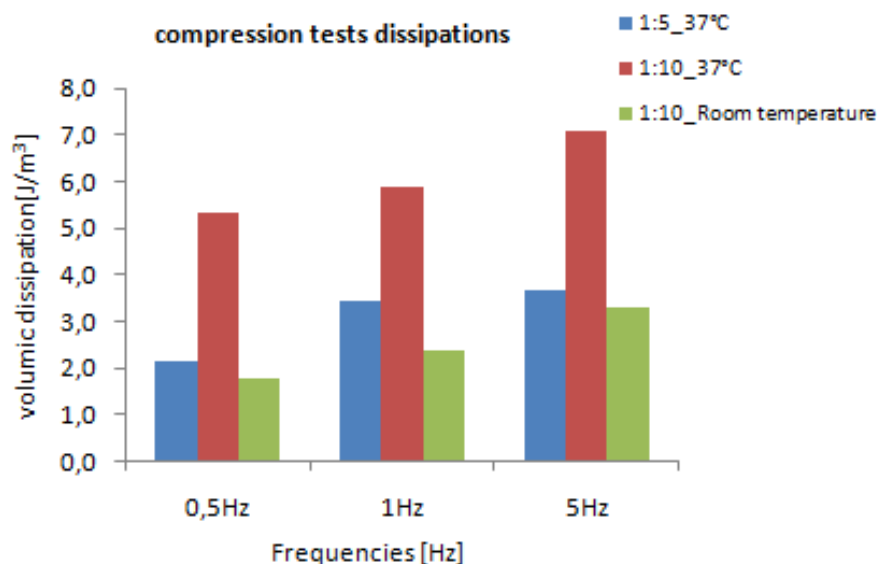


Figure10. Histogram presenting the dissipations of the three chambers tested in compression and at three frequencies

From figure, for the crosslinker effect, the trend observed with the tensile test seems to be confirmed. In fact from to show the effect of the crosslinker, we can compare only the

samples polymerized at the same temperature: 1:5_37°C and 1:10_37°C. 1:5 seems to dissipate less than 1:10 as observed in tensile.

For the polymerization temperature effect, only samples with the same mixing ratio should be compared, thus 1:10_Room temperature and 1:10_37°C. But here rather than the observation made in tensile test, the small increase in polymerization temperature (from room temperature to 37°C) seems to increase the dissipation of the material. This might be due to the thinness of the material membrane on the walls of the encapsulation device. By combining the temperature and the crosslinker effect, the ratio at 1:10 polymerized at room temperature seems to be the less dissipative. But those results are not significant because only one test cycle was performed for each sample

The frequency increase seems to have the same effect on the dissipation as in tensile test. As it increases, the dissipation increases also. And the material seems to dissipate more in chambers walls than in shouldered bars. This might be due to the fact tests performed on the two types of samples are different

CONCLUSION

As the dissipation considerations for materials properties are more and more needed, as the need of materials with less dissipation grows up, and due to the rather limited amount of published materials dissipation data. Our project focused on the characterization of the dissipation of the PDMS (Sylgard 184) for the potential use in fabrication of encapsulation chambers to be used when mechanically testing soft tissue and materials which are difficult to handle such as hydrogels. For this purpose different samples were prepared at different polymerization temperature and different mixing ratios between the Sylgard base and its hardener. Then tensile tests and compression tests were performed on the material.

We observed that the 1:10 ratio which is the recommended one [8] and currently used is the ratio which dissipate the most. By decreasing or increasing the ratio, the material dissipated less energy. Increasing the polymerization temperature increased the dissipation of the material. The increase in strain rate showed also an increase in the dissipation of the material. The encapsulation chamber chambers were difficult to obtain with the used moulds. By now for our chambers and with our moulds the less dissipative chambers made were at the ratio of 1:10 at room temperature. The dissipations obtained with tensile test bar and with chambers were different but showed the same trend for crosslinker and temperature effects. But combining the two effects the less dissipative sample in tensile was 1:5 at 37°C which were different to the less dissipative chamber obtained. Due to the low number of samples used in this project, the observations made seem to be not consistent. They should be verified by testing a large library of samples and computing statistics. Our results are indicatives and we hope that they can serve as bases for further works on the designing of encapsulation chambers. Future research should focus on designing new molds (in another material) for the chambers or finding new materials that could be used in chambers instead of PDMS.

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