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1

Mechanical and microstructural characterization of polyurethane foams

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Abstract. For the manufacture of polyurethane foam having a certain density, we seek to control as much as possible the development of the microstructure. Therefore, the final product could exhibit the desired properties for a notable application in non-lethal projectiles. These last warheads based on these alveolar materials must be able to put out of harm's way a target without causing a permanent injury or a fatal outcome. The mechanical characterization was carried out by dynamic drop weight test, on a machine designed and carried out locally, the mechanical properties of polyurethane foams are highly dependent on density, cell structure (size and shape), and percentage of open and closed cells. The foam may have a preferential orientation in the cell structure. This was followed by a Raman spectroscopy analysis to visualize the semi-opened cells of the cellular polymer. It appears that the cellular polymer has stubborn regular cellular structures with noticeable overlap reversibility.

Keywords: Polyurethane Foam, Drop weight tests, Non-lethal Projectiles, Raman spectroscopy analysis.

1 Experimental methods

1.1 Drop weight tests

The impact tests were carried out according to the recommendations of the NF EN ISO 6603-2 standard. For these dynamic tests, free drop weight of weight is used without initial velocity on a specimen placed at its base. It is equipped with a steel impactor having a square shape. The total mass of the weight drop weight is 5kg and the maximum drop weight height is 1.20 meters. The latter is equipped with a sensor piezoelectric force type PCB 203B, with an acquisition card whose role is to bind the sensor with the digital oscilloscope with a bandwidth of 200MHZ, which is sufficient given the test time and a color screen and a USB input for data backup, to recover the forces exerted during the impact tests as a function of time. These measures are used in the analysis of the evolution of the strength and duration of impact. The configuration of the test is illustrated (Fig. 1).

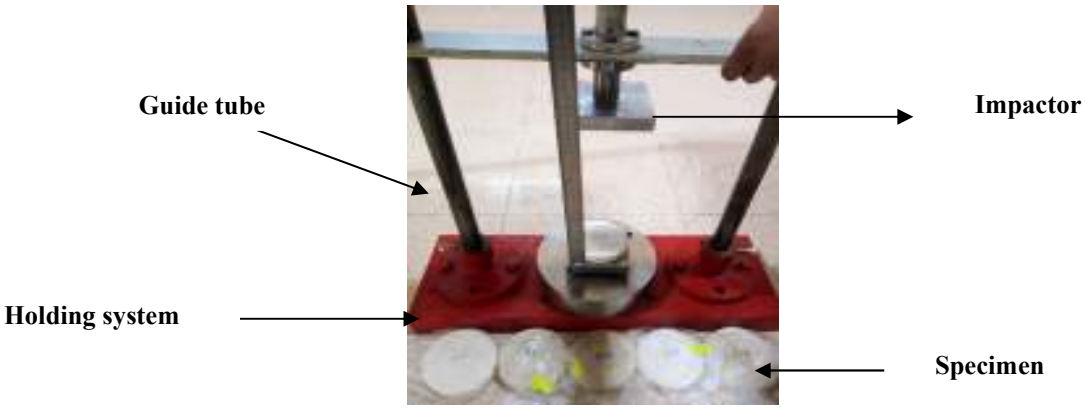

Fig.1. Diagram of the drop weight test.

The cylindrical specimen is about 1 cm long and 10 cm diameter prepared with the same formulations as the hydrostatic compression tests by a special mold for preparing different dimensions of the samples to drop weight tests, to maximize the free drop weight contact area. The specimen is free during an impact according to Standard AITM 1-0010 (Fig. 1).

Table 1. Characteristics of drop weight tests.

Name of the test	Density (g/mm^3)	Impact velocity (ms^{-1})	Deformation speed (s^{-1})
Essays Ep_1	0.43	4.0	0.23
Essays Ep_2	0.15	3.9	0.23
Essays Ep_3	0.27	3.9	0.24
Essays Ep_4	0.30	4.1	0.25
Essays Ep_5	0.22	3.8	0.24
Essays Ep_6	0.38	4.0	0.24

After the preparation, we cut the samples to the milling machine by the rotation of the cutting tool, on the one hand, and the advance of the workpiece on the other hand. The machine is equipped with numerical control, to realize all types of even complex forms.

The free drop weight test is used for laminate impact with a stacking sequence. To obtain some impact energy E_{impact}, the potential energy of the impactor is transformed into kinetic energy. The initial distance "H" between the upper surface of the specimen and the end of the impactor is calculated by:

$$H = 1.1 \frac{\mathrm{E_{impacteur}}}{\mathrm{g} * M_{impact}} \qquad (1)$$

Where g is gravity = 9.81 m / s². The mass of the impactor "$M_{impactor}$" is 5 kg while factor 1.1 is used to compensate for the losses occurring during the test, such as the friction between the guide tube and the assembly of drop weight.

Fig. 2. Arrangement of equipment for an impact test.

In what follows, the elements of the drop weight tests and their respective functionalities are described. The components are shown in (Fig. 3)

Fig.3. blackened flat Impactor of the chess drop weight tests.

For the acquisition of signals, a digital oscilloscope is used. For each input signal, a particular voltage range is set. The test is very fast, it offers a wide range of frequencies. Thus, the frequency range of the acquisition system is set at its maximum of 200 kHz and with each Δt = 0.005 ms the values are recorded. This interval is very short (a few milliseconds), so a trigger, associated with the signal of the force, is used for signal recording. When a certain threshold is exceeded, the data backup is performed. The acquisition system is also able to save pre-trigger data, so all relevant data is available.

The test can be analyzed with the data collected during the impact. The first parameter to be determined is the impact force acting simultaneously on the impactor and the specimen. The force sensor delivers a voltage that is equal to the force in KN, 1V = 1KN.

1.2 Raman spectroscopy

Raman spectroscopy is a complementary technique to infrared spectrometry and is used for the organic and inorganic characterization of polymers and pigments [1, 2]. The Raman spectrum generally shows peaks whose Raman offsets by the wavelength differences with the laser excitation light, characterize the vibration frequencies of the atoms in the molecules present.

Raman spectroscopy is based on the absorption of photons at a specific frequency followed by a diffusion phenomenon at a higher or lower frequency. The modification of scattered photon scattering either by gaining or losing energy results from the vibratory and rotational movement of the molecules in the sample [3, 4].

For the Raman characterization, we have used a Forman 685-2 Foster and Freeman model spectrometer, driven by the Form 685-2 software, with a laser diode emitting red light with a wavelength of 685 nm. Raman offsets are determined in the range 400 - 2000 cm^{-1}. The acquisition parameters used are as follows: 532 nm green laser with a power of 1%, the objective of the x50 microscope, spectral range in wavenumber from 100 to 3200 cm^{-1}, exposure time 10 sec, 3 accumulations for each spectrum (Fig. 4).

2 Results

2.1 Characterization of a Polyurethane Foam by drop weight test:

The drop weight test is a dynamic validation test, particularly about the material behavior laws. The stress results curves are measured as a function of the crushing of the sample (obtained by processing the impactor displacement signal). For a better reading, the data are presented in absolute value. The piezoelectric sensor has a sensitivity of 56.2mv per 1KN, hence the need to process the results obtained from the oscilloscope by Excel to obtain the data. true curves of the evolution of the force as a function of time. **Error! Reference source not found.** represents the evolution of the impact force as a function of time for six impact energies, obtained on six samples of the polyurethane foams. It can be seen that for impact energies lower than 15J, these curves can be assimilated to a sinusoidal shape, two critical points can be distinguished, F0, the second is the maximum force of the impact recorded by the force sensor noted F_{max} is proportional to the initial potential energy or we find a value of 5000N as the maximum value for the energy of the PUR drop weight test Ep 01 and

Ep 03, and about 13000 N for the energy PUR drop weight test Ep 02, and about 5500 N for the energy test PUR lap tumors Ep 05 and Ep 06, and about 3800 N for the energy test PUR lap tumors Ep 04, this is explained by the high kinetic energy during the impact which generates a significant impact force compared to the low energies.

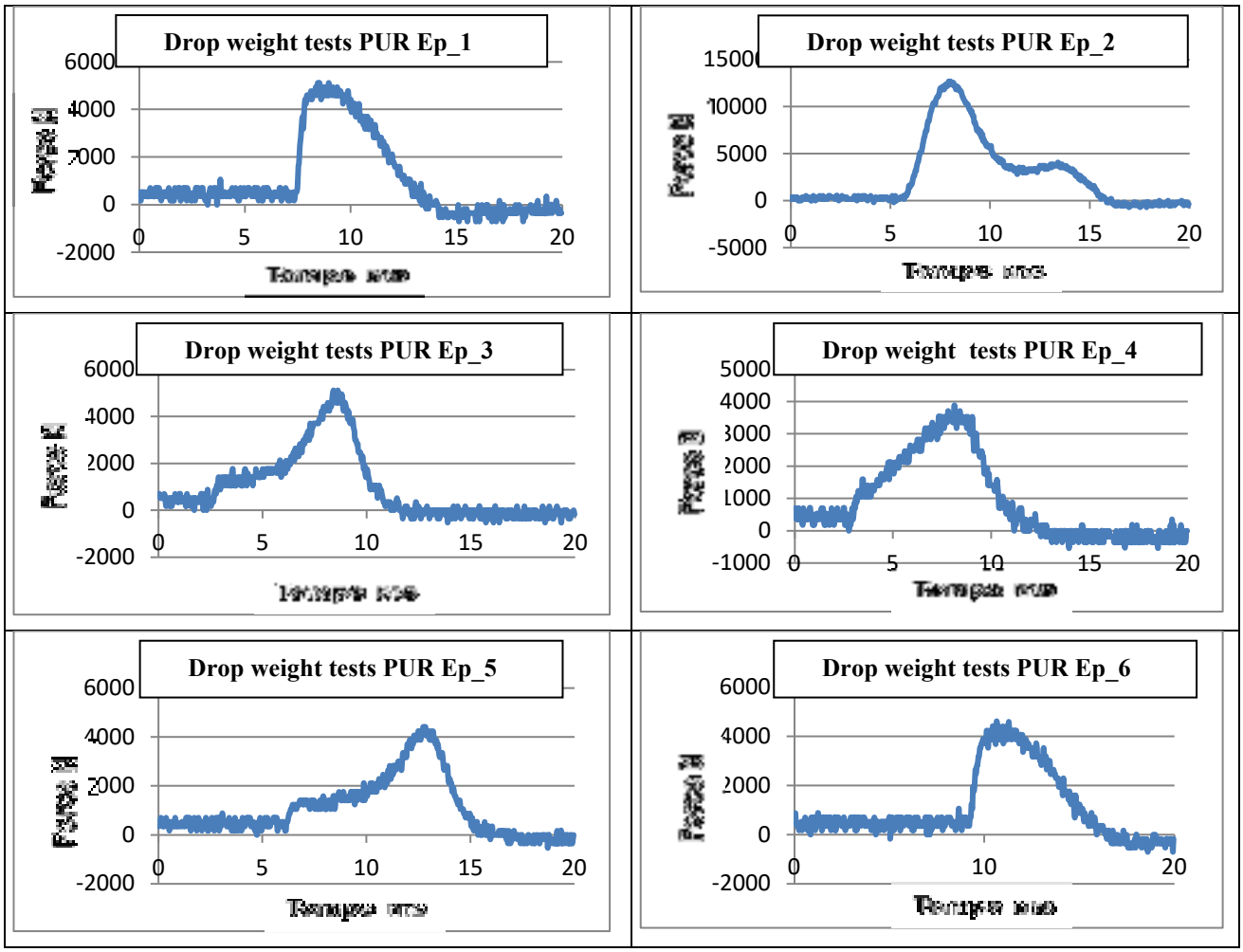

Fig. 4. The characteristic force-time curve of polyurethane foams.

The evolution of the impact force is shown as a function of the displacement. Until a displacement at the beginning of the application of the force on the sample, the force increases linearly with the displacement of the impactor. The stiffness of the polyurethane foam is high. The rigidity of the specimens decreased to around 1500N/ms.

The temporal evolution of the energy transmitted by the impactor to the laminated foam, during the impact test, is illustrated in Fig.4. We can note that the energy of the foam increases to a maximum value equals to the kinetic energy. Based on the first results of the impact tests (force-time curves) associated with the visual inspection of the impacted samples. The different energy and the corresponding velocities are presented in Table 2.

Table. 2. Impact forces and corresponding velocities for specimens

The samples	Ep_1	Ep_2	Ep_3	Ep_4	Ep_5	Ep_6
F_{max} (N)	5000	13000	5000	3800	5500	5500
V (m/s)	0.87	1.9	2.4	3.2	3.4	3.8

Table 2 that the test pieces E_2 has better resistance to impacts concerning the rest of the samples. From the impact velocity values, it is possible to estimate that, theoretically, the initial deformation velocities localized under the impactor (here we consider the rate of deformation of the initial point of contact between the impactor and the sample, thus located on the axis of symmetry of the impactor) are $\dot{\varepsilon}_0 = 170\ s^{-1}$ for a drop height of 330 mm. Fig. 4 shows that, for the tests carried out, the impact velocities had only a small influence on the stress/crush curves obtained, a consistent result gave their re-strewed variation range. However, the results presented are to be taken with great care for crushes greater than 5 mm, i.e. more than 33% of crushing. Indeed, given the variability observed in the results, it seems that the relevance of the proposed curves is no longer acquired.

The set of characterization tests made it possible to highlight the viscosity nature of typical behavior of viscoelastic materials because of its strong dependence on the rate of deformation parameter (in particular the increase in the value of Young's modulus and the response of the material in terms of stress with the increase of $\dot{\varepsilon}_0$).

2.2 Raman spectroscopy

The purpose of Raman spectroscopy analysis is to confirm that the addition of additives always gives rise to a PUR and to affirm, at the same time, the non-existence of side reactions leading to other undesired products as shown in the diagram. next (Fig. 5).

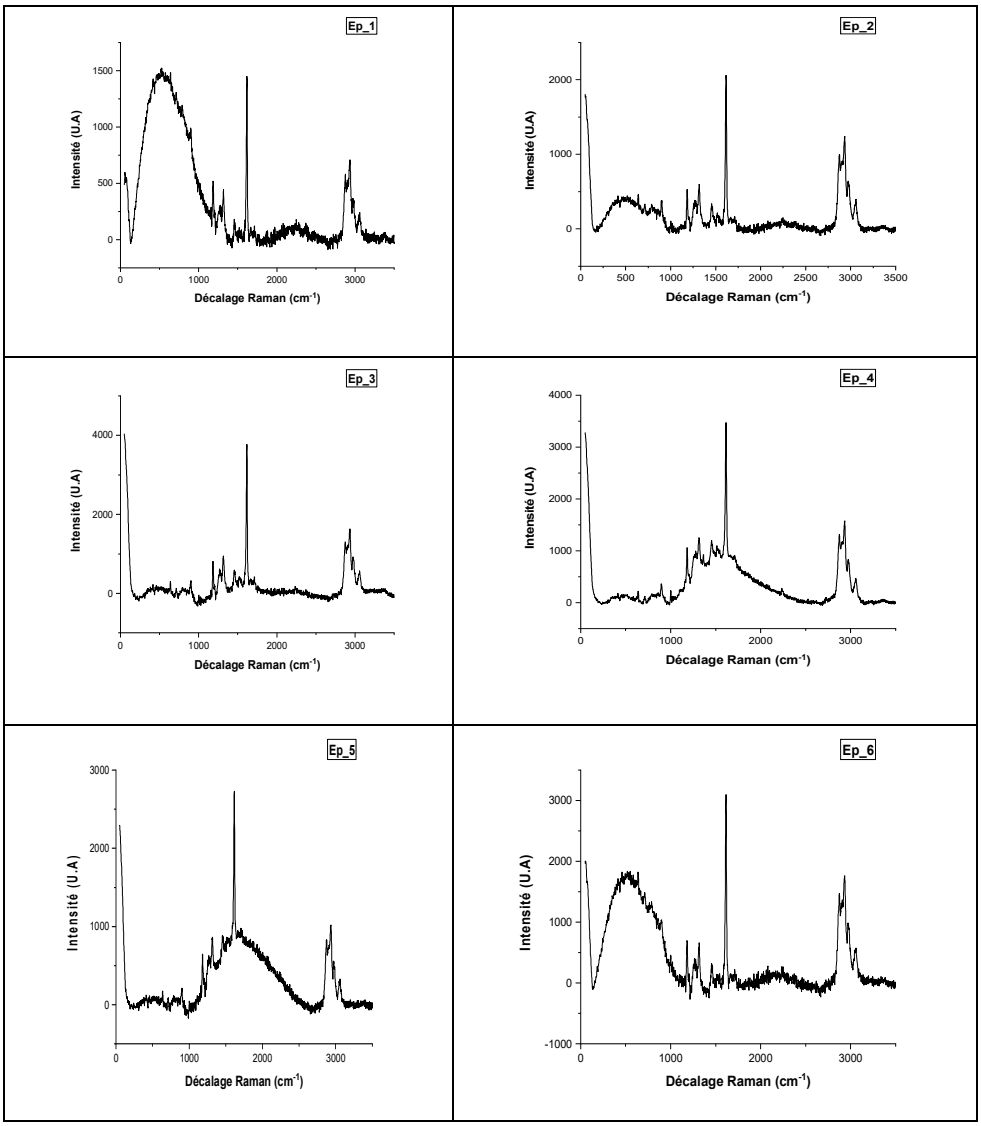

Fig. 5. Raman spectra of different PUR (s) with various additives.

From fig. 5, it becomes clear that the addition of the additives does not in any way change the basic formulation (PUR) and does not give secondary reactions detected by these two spectral techniques. The peak at 1530 is attributed by some [5] to the 4,4'-para-MDI isomers in the MPDI. Others say that it accounts for 1/3 of the C-C elongation vibrations in monosubstituted benzenes (the aromatic rings of PMDI) [6].

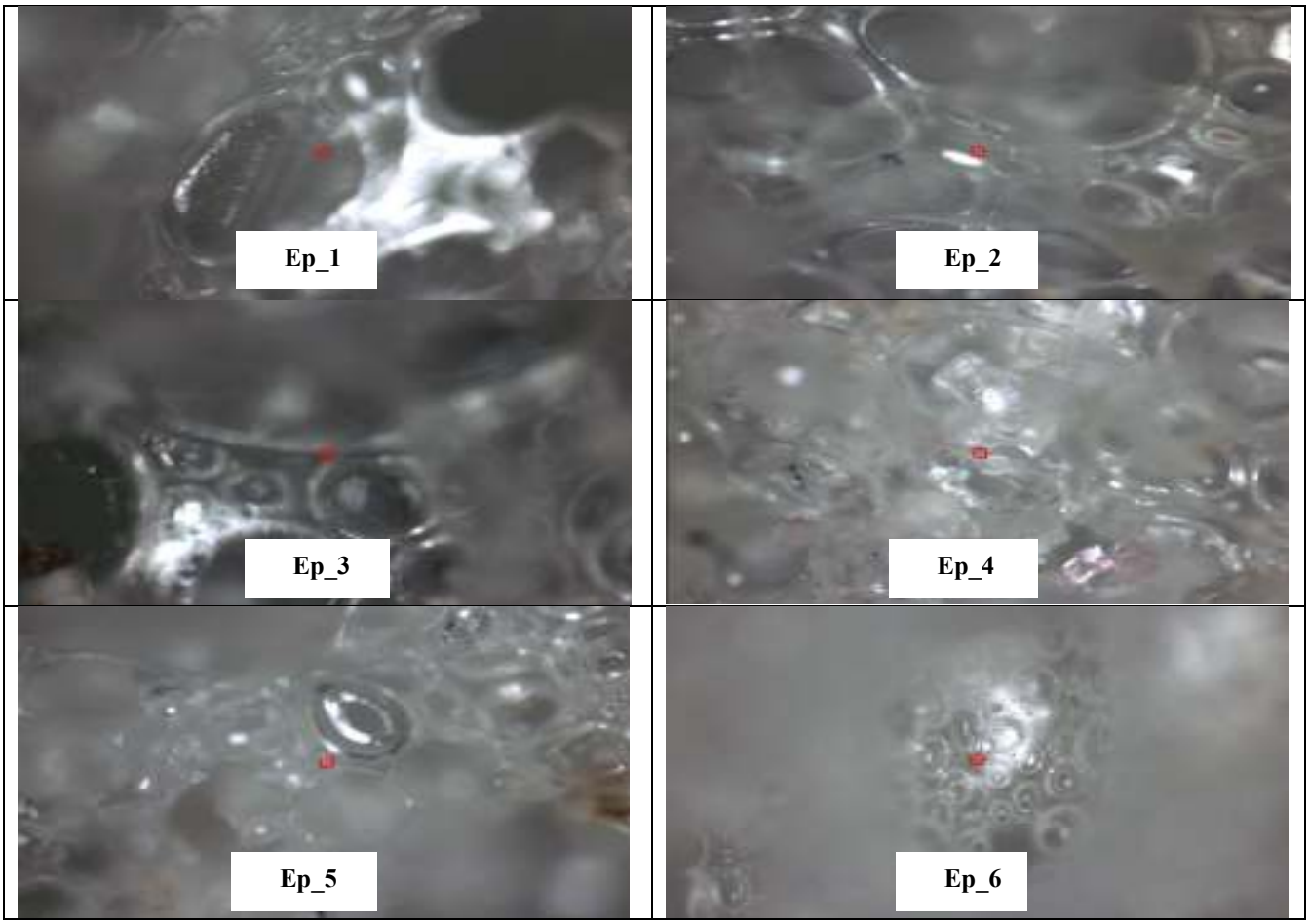

Fig.6. Image obtained by Raman microscope of polyurethane foam

Microstructural characterization: The results of microstructural characterization of the polyurethane foam used as reference material are presented (Fig. 6). The image obtained by the Raman microscope shows a partially open-cell foam not completely closed by its wall and communicating with other cells or with the outside. Cell membranes are visible between the walls of some cells. However, the majority of cells show the absence of membranes. Foam with open or partially open cells is generally a soft or semi-flexible foam. In our case, the partially open cell structure is consistent with the soft nature of the foam.

3 Conclusions

Objectives for the mechanical characterization of polyurethane foams having a certain density. These last warheads based on these alveolar materials must be able to put out

of harm's way a target without causing a permanent injury or a fatal outcome the experimental results of the mechanical tests are presented. Dynamic tests were carried out on polyurethane foam drop weight tests on a machine designed and built locally. Then, a microscopic analysis using a Raman spectroscopy analysis to visualize the semi-open cells of the alveolar polymer is proposed. Because of the results obtained; it appears that the pieces made of the cellular polymer have stubborn regular cellular structures with noticeable overlap reversibility.

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