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REVIEW ARTICLE

Fabrication and Testing of Auxetic Foams for Rehabilitation Applications

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Abstract | Negative Poisson's ratio or auxetic foams were fabricated and studied for use in prosthetic applications. A thermomechanical process was used to convert conventional polyurethane (PU) foam into auxetic PU foam. Two types of auxetic PU foams were made using two different volumetric compression ratios, 3.02 and 3.86. Chemical composition analysis showed that chemical structure of the converted auxetic foam remained the same as the conventional PU foam after the thermomechanical process. Compression tests showed that the auxetic foams had higher stiffness than conventional foams, implying that the auxetic foams could be potentially used in seat cushions, shoe soles, and as liners in prosthetic applications. Compression fatigue tests showed that percentage loss in thickness in auxetic foam fabricated using lower compression ratio had loss in thickness that is comparable to conventional foam, whereas auxetic foam fabricated using higher compression ratio had greater loss in thickness. Our study indicates that the auxetic foams made using lower compression ratio need to be further explored for suitability in various rehabilitation applications.

Keywords: *Cushioning in prosthetics, Negative Poisson's ratio, Compression fatigue, Compression ratio*

1 Introduction

Auxetic materials are negative Poisson's ratio materials. The word auxetic comes from 'auxetikos' which is a Greek word that means "tends to increase". When stretched longitudinally, normal materials contract in lateral direction. Auxetic materials, on the other hand, expand laterally when stretched longitudinally. There are some materials in nature that exhibit auxeticity (negative Poisson's ratio behavior) like cancellous bone, cow teeth skin, and cat skin. Lakes[18] has listed and identified the microstructural features that give rise to negative Poisson's ratio behavior in various materials. There are different geometric patterns such as hexagonal honeycombs, reentrant honeycombs, rotating rigid structures, chiral structures, etc., which give auxetic effect on application of load.[18] Researchers have also discovered various fabrication methods to modify the microstructure of regular materials, such that they exhibit auxetic behavior under certain loading conditions[4, 9, 13, 15, 23]. Auxetic materials have been explored for many applications such as packaging, core materials of sandwich panels,[11] dilator for opening the cavity of an artery during heart surgery,[12] and impact protection devices such as pads, gloves, helmets, and mats.[9] In impact protection devices, auxetic foams are expected to improve comfort and support, and exhibit improved energy absorption for lighter and/or thinner components. In this work, we studied auxetic PU foams for their potential use in prosthetic applications.

PU foams are used in a variety of rehabilitation applications such as mattresses in hospital beds, wheelchair cushions, shoe soles, liners in prostheses, etc. In most of these cases, the purpose of these cushioning materials is to improve comfort and fitting. Improper distribution of forces and stresses can cause pressure sores, blisters, edema, skin macerations, etc.[19, 21] The ability of these materials to enable better stress distribution, air circulation, and stability is critical to ensure functionality and safe operation of the devices/aids. In case of the stump-socket interface

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of a prosthesis user, periodical change in the volume and shape of the residual limb causes loosening and pistoning effect which is detrimental to the function and comfort of the user.[16] Wang et al.[26] developed a prosthetic apparatus that uses auxetic foam layer in the socket of a prosthetic limb. They claim that the foam compensates for the changes in the volume and shape of the residual limb. However, there is no literature, showing that the said apparatus does compensate for the changes. In most of the prosthetic applications, the materials at the user–prosthesis interface are subjected to compressive, shearing, and impact loads. In this work, auxetic PU foams are fabricated and studied using compressive and fatigue tests.

The first man-made auxetic PU foam was introduced by Lakes.[17] Researchers have found that auxetic foams have high indentation resistance,[5] high fracture toughness,[8] synclastic behavior,[11] and good acoustic absorption.[6] Some researchers have also observed shape memory behavior in auxetic foams.[3] Different methods to fabricate auxetic foams from the conventional foams are described in the literature. One of the first methods describing fabrication of auxetic foams from conventional (PU and metallic) foams was given by Lakes.[17] This is achieved by tri-axial compression (using a compression ratio of 1.4:4) to ensure that the cellular ribs permanently protruded inwards. This is followed by heating to a temperature above softening temperature of the foam. Friis et al. [14] also described similar methodology to transform thermosetting, thermoplastic, and metallic foams. While Lakes [17] described the general procedure to make auxetic PU foams, Chan and Evans [4] gave a more detailed description of the process parameters and fabrication technique for both small and large auxetic foam specimens. In their work, they introduced multi-stage method which ensures homogeneous properties in the fabricated foam. Smith et al.[25] showed that the tri-axial compression and heating process can cause the cell ribs to break. They proposed a novel model (missing rib model) to describe the behavior of auxetic foams. Scarpa et al.[23] used a cylinder and piston arrangement for fabrication of auxetic foams. Duncan et al.[9] fabricated auxetic foams using through-thickness pins. They showed that the use of pins provides greater control of in-plane compression. Li and Zeng[20] conducted a wide range of tests and characterizations to study the morphologies, chemistry, and microstructure of three different auxetic foams. They found that the presence of SAN copolymers enabled faster convertibility of PU foams to auxetic foams. Their results also showed that the softening temperature might vary for PU foams depending on the chemical composition and presence of different particles in the foams. Therefore, a clear understanding of materials involved in the manufacture of the raw PU foams is necessary before conversion into auxetic foams. Methods other than thermomechanical process for conversion of PU foams to auxetic PU foams such as chemo-mechanical process,[15] steam-based method for closed cell PU foams[13] are also reported in the literature. In this work, we used the thermomechanical tri-axial compression and heating method for fabrication of auxetic foams.

Very few researchers have conducted fatigue tests on auxetic foams. Scarpa et al.[24] have reported behavior of auxetic PU foam pads for compressive cyclic loading conditions. They found that the damping capacity of the auxetic foam was higher by a factor of 10 when compared to conventional foam. Bezazi and Scarpa[1, 2] showed that auxetic foams have higher mechanical resilience and resistance to failure under tension and significantly higher energy dissipation under cyclic compressive loading. Some researchers have looked at the dynamic, acoustic, and impact characteristics of auxetic foams. Duncan et al.[10] reported quasi-static compression and impact testing on auxetic foams, and reported that auxetic foams reduced the peak force by ten times than conventional foams. Scarpa et al.[22, 23] performed a series of tests to study the dynamic properties of auxetic foams. They found that the auxetic foams showed superior dynamic and acoustic properties when compared to conventional foams. Scarpa et al.[24] found that the transmissibility of auxetic foams fall below 0.6 above 100 Hz, implying that these foams might be ideal for vibration damping applications.

In this work, commercially available PU foams with three different densities were explored for conversion to auxetic foams. Thermogravimetric analysis on the PU foam was performed to determine the softening temperature. Foams that were successfully converted to auxetic PU foam were further studied for compression and fatigue strength. Fourier transform infra-red analysis on both the PU foam and the converted auxetic PU foam was performed to examine the chemical composition before and after the thermomechanical process. Compression and compression fatigue tests were carried out on the auxetic and conventional PU foams.

2 Materials and Methods

PU foams with three different densities (10, 28, and 32 g/cc) were procured from Royal Foam Products, Ranipet, Vellore, India, for making auxetic foams. In this work, a method similar to the two-stage thermomechanical fabrication process[4, 25] was used to fabricate auxetic PU foam from conventional PU foam. Conventional PU foam was tri-axially compressed and heated to a predetermined temperature for a chosen amount of time. The foam was then cooled to room temperature, stretched for 10 min, and then reheated and then cooled back to room temperature.

Initially, thermogravimetric analysis (TGA) was used to determine the optimum heating temperature for each of the foams. The TGA was performed using samples weighing 2.85 mg by heating from room temperature to 750 °C at a heating rate of 10 °C/min in a high pure argon (12 dm^3 h^{-1}) atmosphere (Seiko thermoanalyzer, model SII 7200). The results showed a softening temperature in the range of 200–250 °C for all the three PU foams. Therefore, a heating temperature of 230 °C was used for the fabrication. Two different auxetic PU foams were fabricated using two different compression ratios, 3.02 and 3.86, respectively. PU foam of dimensions $145 \times 100 \times 40$ mm was squeezed into an aluminum box of dimensions $120 \times 80 \times 20$ mm resulting in a volumetric compression ratio of 3.02. The compressed PU foam was then put through a two-stage heating process.[25] Initially, the foam was heated at 230 °C for 25 min in an industrial furnace. Once the foam cooled to room temperature, it was stretched (manually) for approximately 10 min and then reheated at 230 °C for 25 min. This resulted in the conversion of PU foam into auxetic PU foam. Similar process was used for PU foam of dimensions $150 \times 110 \times 45$ mm (volumetric compression ratio 3.86) to obtain auxetic PU foam.

PU foam samples (five samples) of density 32 g/cc consistently showed transition to auxetic PU foam after undergoing the abovementioned fabrication process. On the other hand, PU foams with densities 28 g/cc and 10 g/cc (five samples each) did not show any auxetic behavior after undergoing the same fabrication process (with the same dimensions and compression ratios). Some samples of 28 g/cc and 10 g/cc density foams regained their original uncompressed dimensions after a few days, while others returned to some intermediate dimensions. It is likely that the low-density foams might need different compression ratios and heating times than 32 density PU foams.

2.1 SEM and FT-IR Analyses

Morphologies of the conventional and auxetic PU foam samples were investigated using a ZISS-EVO18 Scanning Electron Microscope (SEM, Carl Zeiss, Germany). Samples were cut into small flat specimens and the surface was sputter-coated with a thin layer of gold before observation. FT-IR spectral analysis has been performed to examine the chemical structure of the foams. FT-IR spectra were recorded for both conventional and auxetic PU foams (obtained using 3.02 and 3.86 compression factors) using an FT-IR spectrophotometer (Shimadzu Crop Iraffinity-1) utilizing a KBr pellet technique with wavenumbers varying from 400 to 4000 cm^{-1} with 30 scans per sample at a resolution of 4 cm^{-1}.

2.2 Compression Tests

Compression tests were conducted on conventional and auxetic PU foams using a Tinius Olsen H5KS universal testing machine with a load cell of 5000 N, as shown in Fig. 1. Foams were compressed at a strain rate of 0.0166 mm/s until it reached a strain of 90%. Samples with dimensions of $50 \times 50 \times 20$ mm^3 were used for performing compression tests.

2.3 Compression Fatigue Tests

The fatigue analysis has been performed on conventional PU foam and auxetic PU foams using a variable loading with sinusoidal waveform with 5 Hz of pulsation. Compression fatigue tests were conducted based on IS 7888 standard using an Instron 8874 fatigue machine at CIPET, Guindy, Chennai. The samples were initially positioned under load condition and then load was applied up to maximum of 75% displacement from initial position (0–15 mm displacement), as shown in Fig. 2. The maximum load (corresponding to 15 mm displacement) was fixed based on static compression tests. All tests were conducted at room temperature with no specific humidity control. Losses of percentage in thickness are calculated after 20,000 cycles of loading.

3 Results and Discussion

Researchers have reported softening temperatures in the range of 170–250 °C for PU foams. However, Li and Zeng[20] have shown that the chemistry and microstructure of starting PU foam could significantly influence the process parameters in conversion of PU foam to auxetic PU foam. Therefore, TGA was performed to assess the thermal stability of flexible PU foam and to find its

Figure 1: Compression test setup.

softening temperature. It was found from the analysis (Fig. 3) that there is a percentage reduction of weight after a temperature of around 200 °C. This weight reduction indicates softening of foam in the temperature range of 200–250 °C. Based on this thermal analysis, a temperature of 230 °C was chosen for the heat treatment to convert conventional PU foam to auxetic foam.

Poisson's ratios of the conventional and auxetic PU foams were measured manually (using method described in [7]). The conventional foam had a positive Poisson's ratio of 0.38 and the fabricated auxetic foams (five samples each) showed Poisson's ratios in the range of − 1.09 to − 1.21 (foam fabricated using 3.02 compression ratio) and − 1.36 to − 1.48 (foam fabricated using 3.86 compression ratio). The Poisson's ratio was determined at a longitudinal elongation of 4 mm.

FT-IR analysis of the conventional and auxetic PU foams showed very similar spectra (Fig. 4), indicating that the chemical bonds remained intact after the heat treatment at softening temperature. The peaks observed in PU and auxetic foams are—a peak at 3278.6 cm^{-1} corresponding to N–H stretching, peaks at 2970.3 and 2866.28 cm^{-1} corresponding to C–H stretch, a

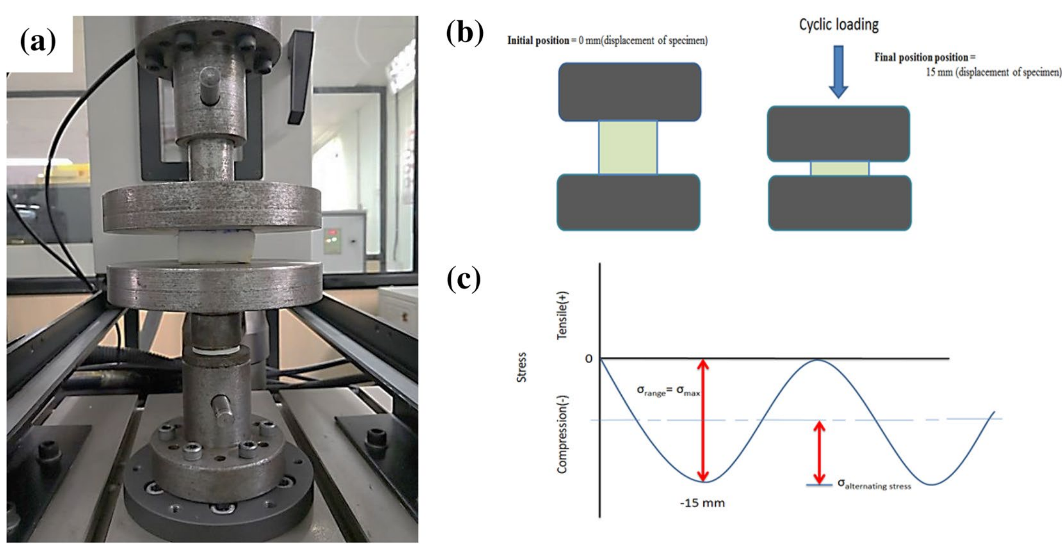

Figure 2: **a** Fatigue test setup at Advanced Research School for Technology & Product Simulation, CIPET, Guindy, Chennai. **b** Schematic representation of cyclic loading and **c** graph showing the sinusoidal waveform of the stress applied on the foam in compression fatigue test.

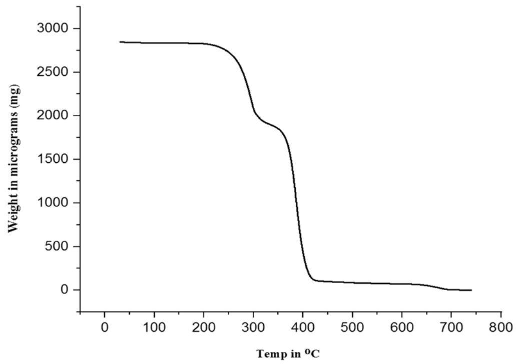

Figure 3: Temperature (in °C) versus weight (TG micro grams) of conventional PU foam (32 g/cc density) obtained from thermogravimetric analyzer.

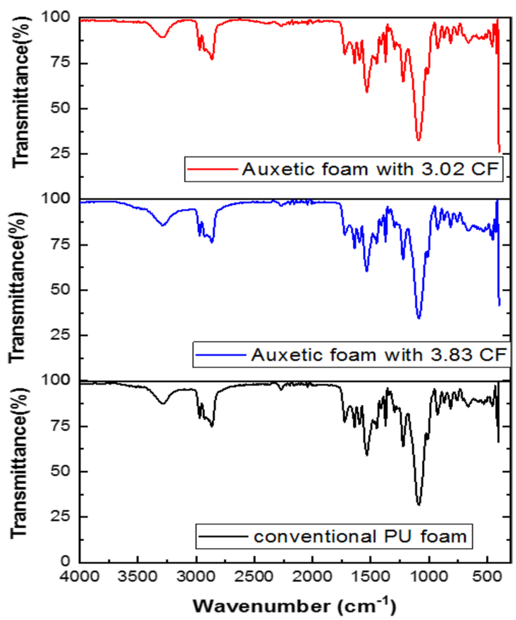

Figure 4: FT-IR spectra for auxetic and conventional polyurethane foam.

peak at 1724.6 cm^{-1} corresponds to C=O groups, a peak at 1533.1 cm^{-1} corresponds to N–H bending, and a peak at wavenumber of 1087.85 cm^{-1} due to C–O–C-stretching vibrations.

Morphology of conventional and auxetic PU foams was studied using SEM. It is evident from the SEM images (Fig. 5) that ribs are broken in the case of auxetic foam (Fig. 5b). This is similar to the results reported by others[3, 15, 20, 25]. The breakage of ribs can be attributed to the tri-axial compression during the process of auxetic foam conversion.

Compression tests were performed and corresponding stress–strain curves were plotted (Figs. 6 and 7). Under compression, conventional foams exhibited linear stress–strain relationship due to compression followed by a plateau region. A magnified view of this trend is depicted in Fig. 7. Auxetic foams show higher stiffness values compared to conventional foams and no plateau regions were observed in the stress–strain curve. Similar trends in stress–strain relationships were observed in the case of both the auxetic PU foams with volumetric compression factors of 3.02 and 3.86. Of the two auxetic PU foams, the one fabricated with higher compression factor of 3.86 showed higher stiffness. This difference in stiffness can be attributed to the higher density of ribs in foam with higher volumetric compression ratio. Similar trends were reported by others[4, 10] where the auxetic foams showed increase in strength with an increase in strains unlike conventional foam.

Fatigue analysis was performed by applying compressive cyclic loads varying from zero stress level to stress values corresponding to 75% of strain for each sample. Fatigue resistance of conventional and auxetic foams was compared based on the percentage loss in thickness after applying pulsating loads for 20,000 cycles at a frequency of 5 Hz. Stresses of 4.94 kPa, 18.75 kPa, and 27 kPa were used in the fatigue test to achieve maximum displacement corresponding to 75% strain for conventional, auxetic foams made using 3.02 CF and auxetic foam made using 3.86 CF, respectively. The obtained values of the percentage loss in thickness for three types of foams are shown in Table 1.

After the fatigue test, auxetic PU foams showed greater percentage of loss in thickness compared to the conventional foams. The triaxial compression and heating process causes the breakage of ribs and weakens the connectivities in the microstructure of the auxetic foams (Fig. 5). Applying a cyclic load on the auxetic foam could have caused further damage of the weakened ribs, thereby causing more loss in the thickness than in the conventional foam. Auxetic foam that was manufactured with larger compression ratio (of 3.86) showed more loss in thickness possibly due to the presence of more number of broken ribs. Compression fatigue tests by Bezazi and Scarpa[1] also showed greater loss in rigidity in auxetic foam possibly due to larger number of broken and/or weakened ribs.

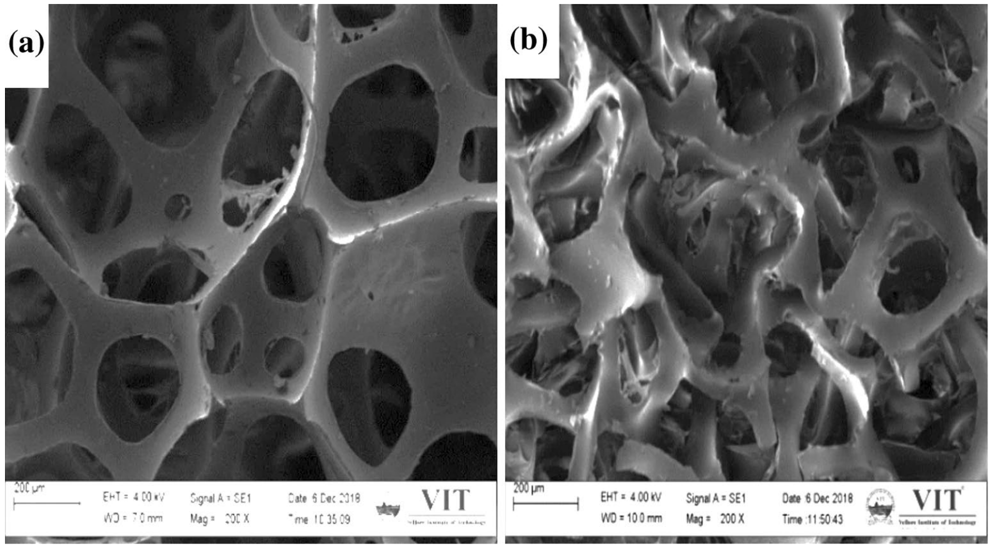

Figure 5: SEM images (200 ×) for **a** conventional polyurethane foams and **b** auxetic polyurethane foam.

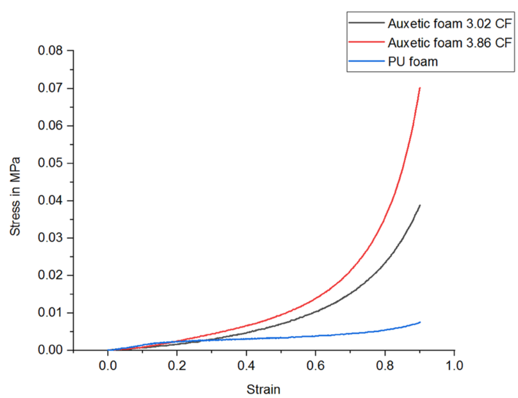

Figure 6: Stress–strain curves for conventional and auxetic polyurethane foams.

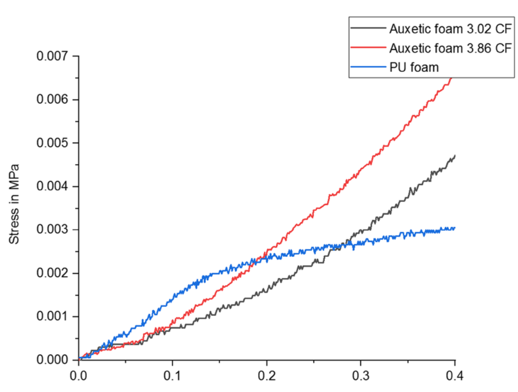

Figure 7: Magnified view of low strain region from stress–strain curves.

Table 1: Results showing percentage loss in thickness in the conventional and auxetic foams (fabricated using 3.02 and 3.86 compression ratio) after compression fatigue test.

Sl. No	Type of sample	Percentage loss in thickness (%)
1	Normal PU foam	1.74
2	Auxetic foam 3.02 CF	2.0
3	Auxetic foam 3.86 CF	4.17

4 Conclusion

Conversion of low-density (10 gm/cc and 28 g/cc) PU foams into auxetic foams was unsuccessful. Although the morphology of the converted low-density foams showed breakage of ribs, the foam samples did not show auxetic behavior. Moreover, the foams returned to their uncompressed dimensions after a few days. It is likely that the number of broken ribs was not high enough to ensure auxeticity and/or the heating time required to set the foam in auxetic state could have been different for low-density foams. Further studies are required to establish the relationship between the density of the raw PU foam and the process parameters in the thermomechanical process of fabricating auxetic foams.

The results from compression show that the auxetic foams are stiffer than conventional PU foams at larger strains and could possibly be used in rehabilitation applications. The compression fatigue results show that auxetic PU foams fabricated using lower compression ratios have similar loss in thickness as the raw foams. Bezazi and Scarpa [1] showed that energy dissipation by auxetic PU foams is significantly higher than conventional foams. Therefore, PU foams fabricated using lower compression factors could be ideal for load-bearing applications such as stump–socket interface. However, other factors such as energy absorption, dissipation, and hysteresis should be examined through proper tests before using these foams for prosthetic applications.

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