

**DEVELOPMENT OF SELF-ACTUATED AUXETIC
STRUCTURE FOR BIOMEDICAL APPLICATION.**



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DEVELOPMENT OF SELF-ACTUATED AUXETIC STRUCTURE FOR BIOMEDICAL APPLICATION.

A thesis submitted in partial fulfillment of the requirement for the degree of

Masters of Sciences

In
Biomedical Sciences

By

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DEDICATION

I dedicate this piece of work to my parents and family members who supported me
in my studies as well as in every matter of my life.

ACKNOWLEDGEMENT

In the name of ALLAH, the most Gracious and the most Merciful, all praise are for Him. He is the one who blessed man with Wisdom and Knowledge. I shower my thanks to Almighty Creator for flourishing my thoughts and helping me in my all efforts to complete this research work.

Many Darood on the Last Prophet Muhammad (peace be upon him) whose teachings have always been source of light in journey of life.

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May Allah bless the above mention personalities with honor and success, Ameen.

Shakra Ahmad

ABSTRACT

Structures having negative Poisson's ratio are known as auxetic structures. These structures expand laterally when stretched longitudinally. Negative Poisson's ratio enhances the mechanical properties of the material like increased plane strain fracture, increased shear modulus, indentation resistance and fracture toughness. These properties can be very useful in medical devices formation. Taking this in consideration micro scale auxetic membrane was developed. For self-actuation, hydrogel was attached to the auxetic membrane. The purpose of self-actuated auxetic structure was to achieve controlled porosity. This micro scale actuation can be used for biomedical applications like controlled drug dispensation and auxetic stents.

TABLE OF CONTENTS

DECLARATION.....	v
COPYRIGHT STATEMENT	vi
ACKNOWLEDGEMENT	viii
ABSTRACT	ix
TABLE OF CONTENTS.....	x
LIST OF FIGURES	xii
LIST OF TABLES	xiv
LIST OF ACRONYMS.....	xv
CHAPTER 1 INTRODUCTION.....	1
1.1. History	1
1.2. Natural auxetic materials:	2
1.3. Manufacture of auxetic structure:	2
1.4. Applications of auxetic materials	3
1.5. Aim of the research	4
Chapter 2 REVIEW OF LITERATURE	5
2.1. Properties of Auxetic materials.....	5
2.2. Types of Auxetic structures	6
2.3. Applications of Auxetic structures.....	10
CHAPTER 3 MATERIALS AND METHODS.....	14
3.1. Materials.....	14
3.1.1. Auxetic membrane	14
3.1.2. Device Skeleton.....	14
3.1.3. Hydrogel (self-actuator).....	14
3.1.4. Software	14
3.2. Methods	15
3.2.1. Fabrication of auxetic film	15
3.2.2. Device skeleton fabrication.....	16
3.2.3. Hydrogel synthesis.....	17
3.2.4. Hydrogel compositions.....	18

3.2.5. Fabrication of self-actuating device.....	19
3.2.5. Validation of the self-actuating system.....	19
3.2.6. Auxetic film expansion analysis	20
3.2.7. Mechanical testing.....	21
CHAPTER 4 RESULTS AND DISCUSSION.....	23
4.1. Hydrogel compositions expansions	23
4.2. Measurement of auxetic film expansion	24
4.3. Mechanical testing	26
CHAPTER 5 CONCLUSION AND FUTURE PERSPECTIVES.....	29
5.1. Conclusion.....	29
5.2. Future prospective.....	29
CHAPTER 6 REFERENCES	30

LIST OF FIGURES

Figure 1. 1: Comparison of conventional and auxetic structure. (Mir et al., 2014).....	1
Figure 1. 2: Scale of auxetic structures miro to molecular level. (Alderson, 1999).	3
Figure 1. 3: Hypothetical Model (Top view)	4
Figure 1. 4: Hypothetical Model (Bottom view).....	4
Figure 2. 1: Auxetic and non-auxetic material showing indentation resistance.	5
Figure 2. 2: Saddle-shaped surface and dome-shaped surface of conventional and auxetic material respectively (Evans and Alderson, 2000).....	6
Figure 2. 3: Honey comb auxetic structure (Mir et al., 2014).....	7
Figure 2. 4: PTFE microstructure at rest and at tensile stress (Mir et al, 2014).	7
Figure 2. 5: Rotating square geometry (Mir et al, 2014).....	8
Figure 2. 6: Rotating parallelogram geometry (Mir et al, 2014).	8
Figure 2. 7: Rotating square geometry with different sizes of squares (Mir et al, 2014).	9
Figure 2. 8: Keyed brick auxetic structure (Evans and Alderson 2000).....	10
Figure 2. 9: Dilator to open small vessels (Bhullar et al., 2015).	10
Figure 2. 10: Drug delivering auxetic stent (Bhullar et al., 2015).	11
Figure 2. 11: (a) Piezoelectric matrix. (b) Unit of piezoelectric matrix (Evans and Alderson 2000). ..	11
Figure 2. 12: Smart bandage releasing drug on wound swelling (White and Liz 2009).	12
Figure 2. 13: Macro model of smart bandage for controlled drug delivery and porosity (Mir et al., 2017).....	13
Figure 3. 1: Rotating square auxetic single unit CAD Design.	15
Figure 3. 2: Auxetic film CAD design.	15

Figure 3. 3: Fabricated auxetic film.....16

Figure 3. 4: (A) Device skeleton base. (B) Constraining clamp top view. (C) Constraining clamp bottom view.16

Figure 3. 5: (A) 3D printed PLA device skeleton base. (B) 3D printed constraining clamp top view. (C) 3D printed constraining clamp bottom view.17

Figure 3. 6: Fabrication of self-actuated auxetic device.....19

Figure 3. 7: Validation of self-actuating system.....19

Figure 3. 8: Flow chart of auxetic film expansion analysis.20

Figure 3. 9: Known length piece for scale.21

Figure 3. 10: Mechanical testing machine.22

Figure 3. 11: The process of mechanical testing.22

Figure 4. 1: Hydrogel expansion graph.24

Figure 4. 2: Images of self-actuated auxetic system with open pores.26

Figure 4. 3: Stress-Strain graph of mechanical testing26

Figure 4. 4: Force-Displacement graph of mechanical testing28

LIST OF TABLES

Table 3. 1: Different hydrogel compositions.	18
Table 4. 1: Different hydrogel compositions and their swelling behavior.....	23
Table 4. 2: Expansion measurement of the membrane.....	25
Table 4. 3: Conventional polymers and their elastic modulus.	27

LIST OF ACRONYMS

Micrometer	um
Millimeter	mm
High molecular weight polyethylene	UHMWPE
Poly lactic acid	PLA
Poly (vinyl alcohol)	PVA
Polyurethane	PU
Polytetra-flouroethylene	PTFE
Polypropylene	PP

CHAPTER 1 INTRODUCTION

The negative ratio of the transverse strain to the axial strain in the direction of loading is defines as Poisson's ratio. Usually stretching causes material to get thinner. This refers to positive Poisson's ratio. is expected to make a material thinner and compressing results in bulge. However, auxetic materials exhibit very unusual property of becoming wider when stretched and narrower when compressed. This is due to negative Poisson's ratios (Liu and Hu, 2010).

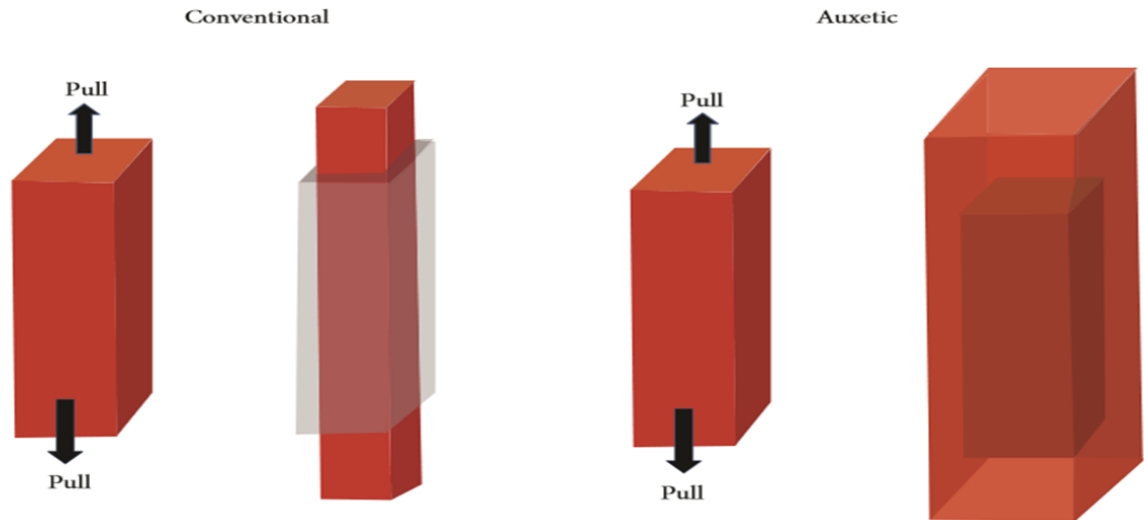


Figure 1. 1:Comparison of conventional and auxetic structure. (Mir *et al.*, 2014).

The mechanisms of auxetic materials depends on either micro or geometrical structure is cause of auxetic behavior of the material. Auxetic mechanism also depends on co- operation between material's deformation and load. Auxeticity is induced in materials through structural design (Bhullar *et al.*, 2015).

1.1.History

Auxetic materials have been known for over 100 years. In early 1900s, Woldemar Voigt was the first physicist to report this property and he observed that the somehow crystals when

longitudinally stretched, become thicker laterally. In 1927, A. E. H. Love presented an example of a crystal pyrite having Poisson's ratio of -0.14. In 1982 Gibson found auxetic effect in two-dimensional silicone rubber and deformed aluminum honeycombs.

Auxetic polyurethane foam was fabricated by Roderick Lakes in 1987. Its commercial availability matured the international concept of auxetic materials and was a major breakthrough in inducing auxeticity in materials through their design (Bhullar *et al.*, 2015).

1.2.Natural auxetic materials:

Naturally, auxetic materials exist in the form of single crystals of arsenic, cadmium, cristobalite and elemental metals having cubic unit structure (Bhullar *et al.*, 2015). However, some biological materials also found to be auxetic. Certain types of skin act as auxetic material, for example cow teat skin, salamander skin, and cat skin (Frolich *et al.*, 1994). In human shins cancellous bone which bear the load also found to be auxetic (Liulan *et al.*, 2007).

1.3.Manufacture of auxetic structure:

The unique abilities of auxetic material open the doors of effort in its made and finding new applications. In view of this, a significant effort has been made towards designing these materials and finding its new applications. Moreover, all major classes of materials have been used for synthesis of auxetic material according to the need. The production of synthetic auxetic materials ranges from macro to nanoscale and to molecular level (Bhullar *et al.*, 2015).

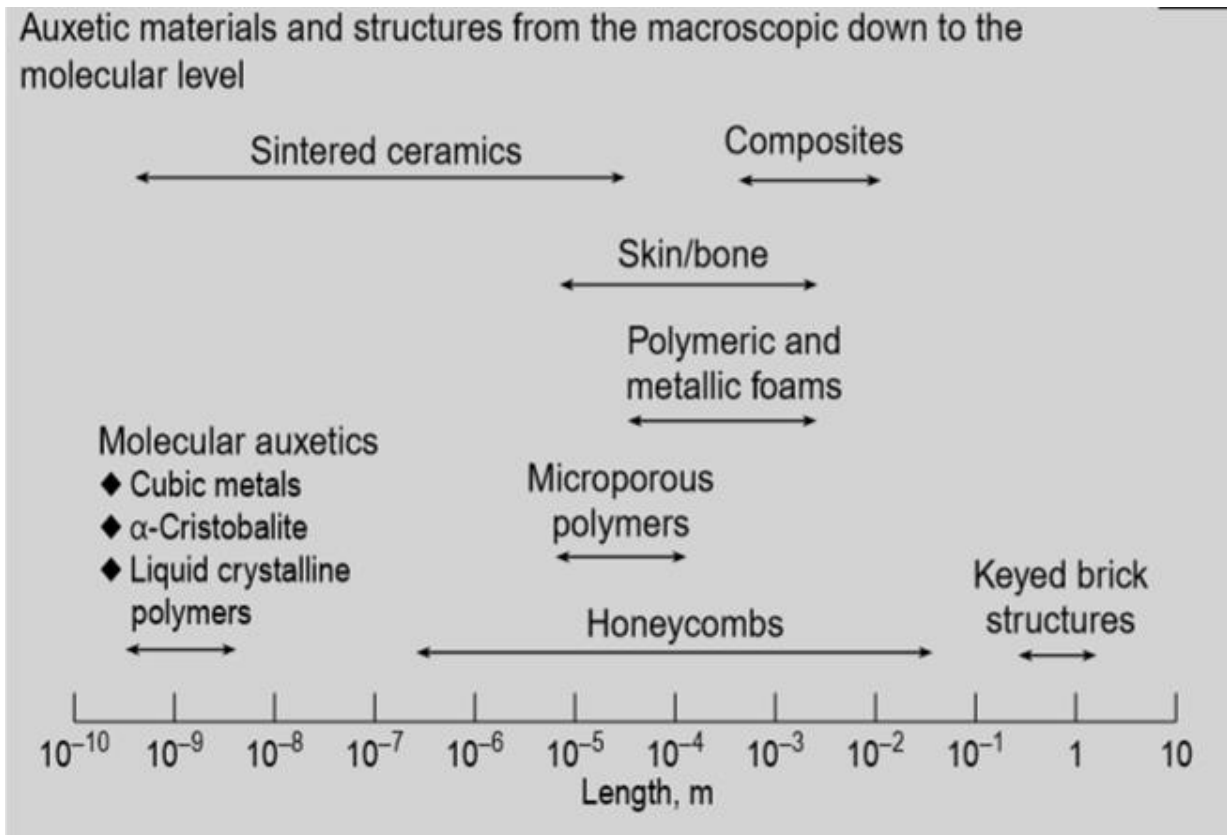


Figure 1. 2: Scale of auxetic structures miro to molecular level. (Alderson, 1999).

1.4.Applications of auxetic materials

Auxetic materials have wide range of applications. It can be used in electromagnetic launcher technology, auxetic body armor, thermal protection, aerospace and automotive applications and biomedical applications e.g. tissue engineering, scaffolds, stents, implants and other medical devices (Evans and Alderson 2000).

1.5.Aim of the research

Aim of current research is to development of Micro-scale auxetic system with following attributes

- Multidimension expansion.
- Self-actuated
- Controlled porosity

The hypothetical model of the system is below.

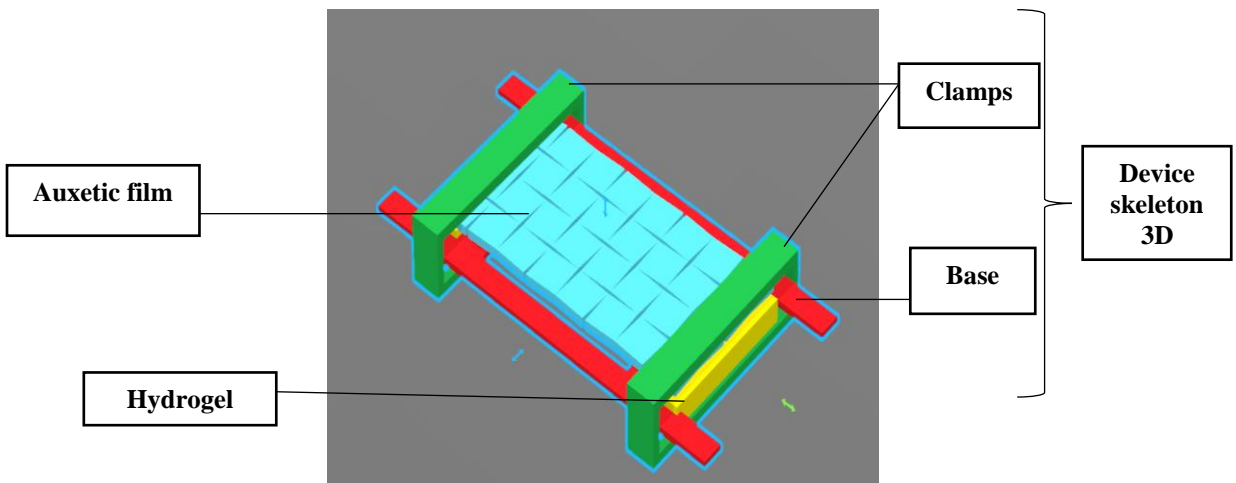


Figure 1. 3: Hypothetical Model (Top view)

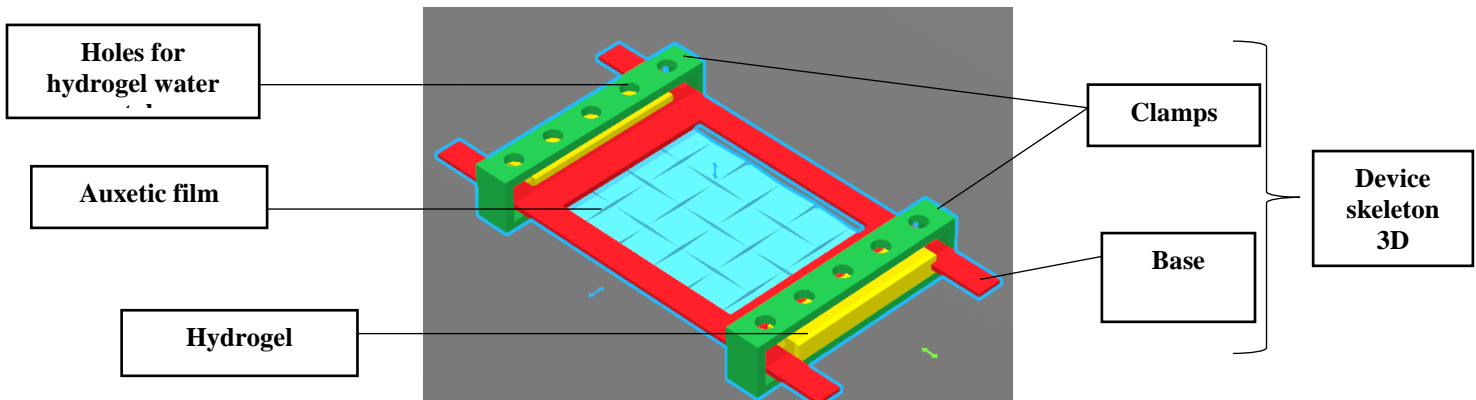


Figure 1. 4: Hypothetical Model (Bottom view)

Chapter 2 REVIEW OF LITERATURE

Biomaterial field have evolved from past years and smart biomaterial have been the reason of it. They are biomimetic in nature. Auxetic materials are included in this list. They are shape memory most of the time and have revolutionized the biomedical industry. They exhibit synclastic behavior (Bhullar *et al.*, 2015).

2.1. Properties of Auxetic materials

With comparison to common materials auxetic materials show unique properties. When a material is turn auxetic its certain mechanical properties enhance such as increased shear modulus, increased plane strain fracture resistance, fracture toughness, indentation resistance and acoustic response. (Yang *et al.*, 2004).

When auxetic material are deformed they exhibit novel behavior. In conventional materials when an object impact it flows the impact laterally causing less indentation resistance of the material. While auxetic material the impact flows into lateral direction enhancing its indentation resistance. It is shown in Figure 2.1 (Evans and Alderson., 2000).

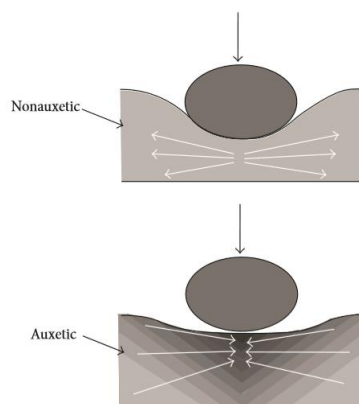


Figure 2. 1: Auxetic and non-auxetic material showing indentation resistance.

Auxetic materials have negative Poisson's ratio. This gives benefit to the material and cause increase in shear stiffness, increased indentation resistance and increased plane strain fracture toughness. Having these properties make auxetic materials better than conventional materials (Grima and Evans 2000).

Auxetic materials have tendency to form synclastic surface instead of saddle shaped surfaces like conventional materials. (Evans and Alderson 2000).

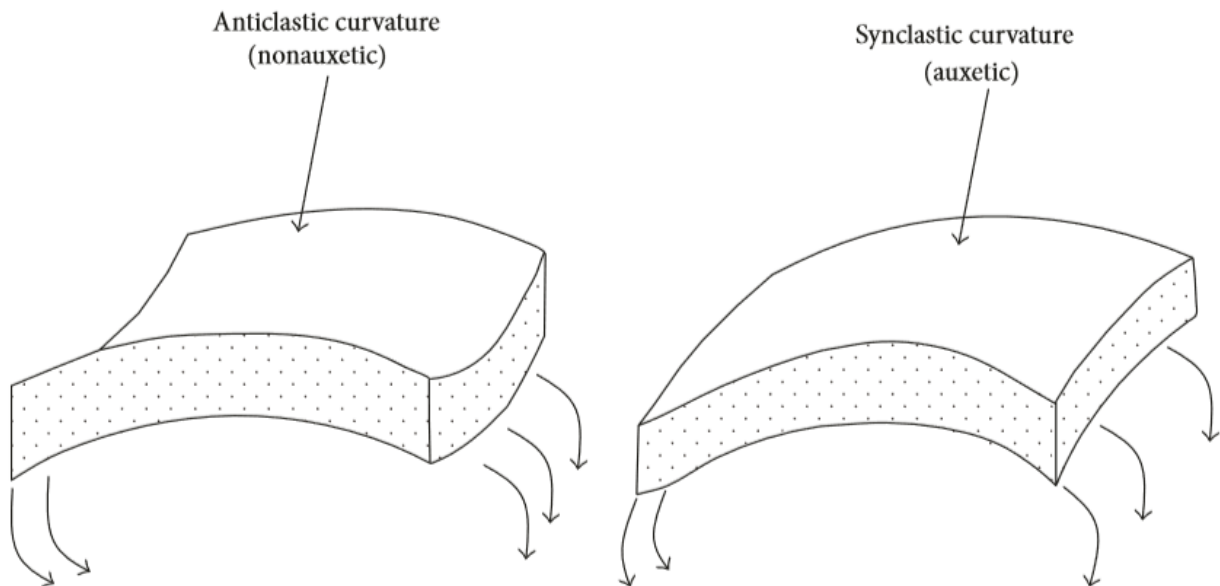


Figure 2. 2: Saddle-shaped surface and dome-shaped surface of conventional and auxetic material respectively (Evans and Alderson, 2000).

Auxetic materials has sound absorbing properties. this make them useful in civil and military application. They are also act as biomimic and act like spongy bone (Stavroulakis, 2005).

2.2. Types of Auxetic structures

Different types of auxetic structure are used now a day. They differ from each other by deformation mechanism and fabrication. Polyurethane and polyethylene foams are used commercially. Apart from these Polytetra-flouroethylene (PTFE), polypropylene (PP) and high

molecular weight polyethylene (UHMWPE) are also used in different industries. Zeolites and α -cristobalite auxetic are naturally occurring auxetic structures (Yang *et al.*, 2004).

The common auxetic structure used is honeycomb structures. It can be easily fabricated. Polymer dies or laser cut technology can fabricate the honeycomb structure (Yang *et al.*, 2004).

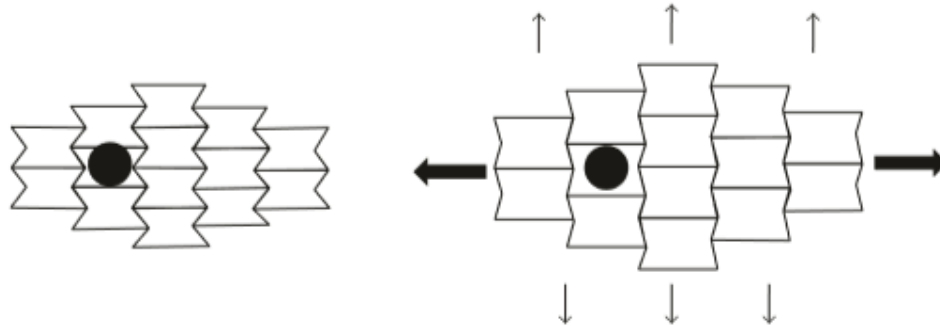


Figure 2. 3: Honey comb auxetic structure (Mir *et al.*, 2014).

Nodules with interconnected fibrils make the microstructure of microporous PTFE microstructure. PTFE has intrinsic properties but the auxetic behavior is due to its microporous structure. It is due to fibrils and nodules cooperative interaction causing the expansion in transverse as well as longitudinal direction (Mir *et al.*, 2014).

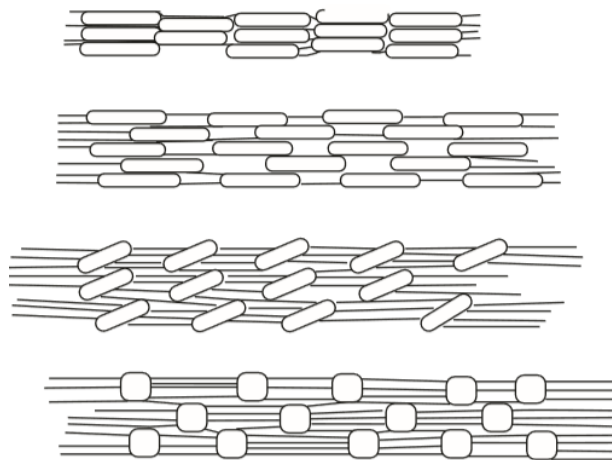


Figure 2. 4: PTFE microstructure at rest and at tensile stress (Mir *et al.*, 2014).

Apart from nodules and fibrils geometric structures with specific arrangements also show auxetic properties. one of these arrangements involve rigid square with hinges as connection between them. One cell is composed of four squares (Mir *et al*, 2014).

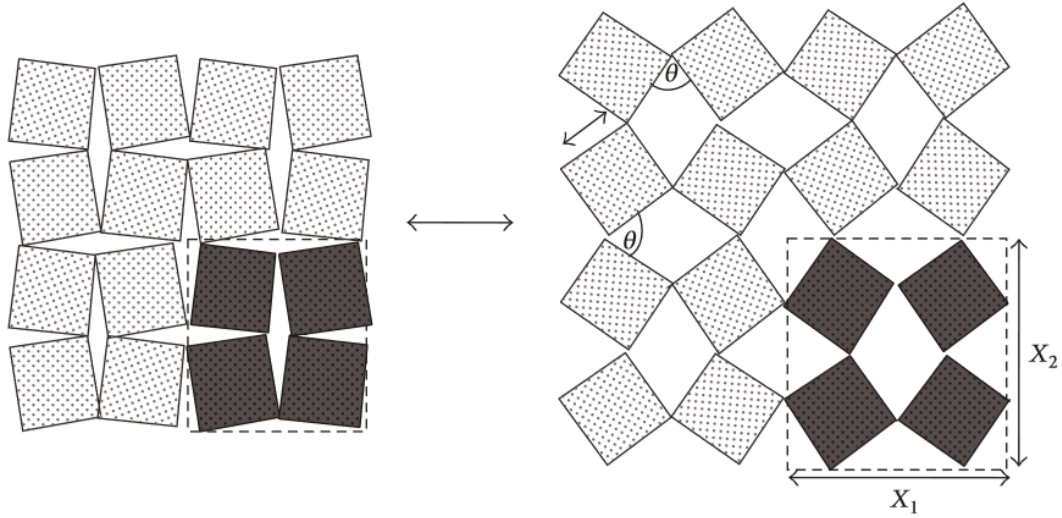


Figure 2. 5: Rotating square geometry (Mir *et al*, 2014).

On the principle of rotating square geometry rotating parallelogram structures are also formed.

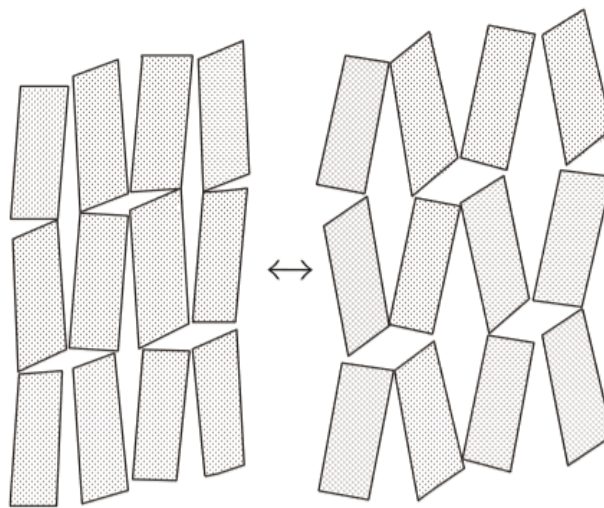


Figure 2. 6: Rotating parallelogram geometry (Mir *et al*, 2014).

Geometrical auxetic structures can also be made by different sizes geometrical shapes to increase the auxeticity.

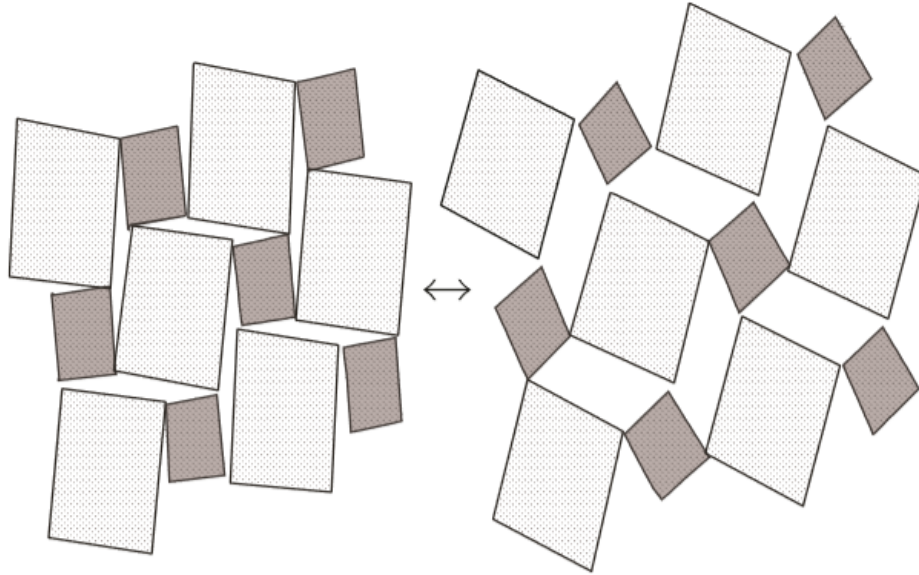


Figure 2. 7: Rotating square geometry with different sizes of squares (Mir *et al*, 2014).

Laser techniques were used to fabricate auxetic polymers like honeycomb auxetic structures. Size of a single unit was $\sim 1\text{mm}$. These micro auxetic structures can be used in passive and active filter membranes. The unique auxetic pore structure help in size selectivity for filtration (Evans and Alderson 2000).

Another type of auxetic structure is Magnox reactor radially keyed graphite moderator core. There are graphite bricks which are loosely connected by corner keys. These keys help in the opening and closing of the structure when stress is given. This structure has low resistance to volume change while high resistance to shear deformation (Evans and Alderson 2000).

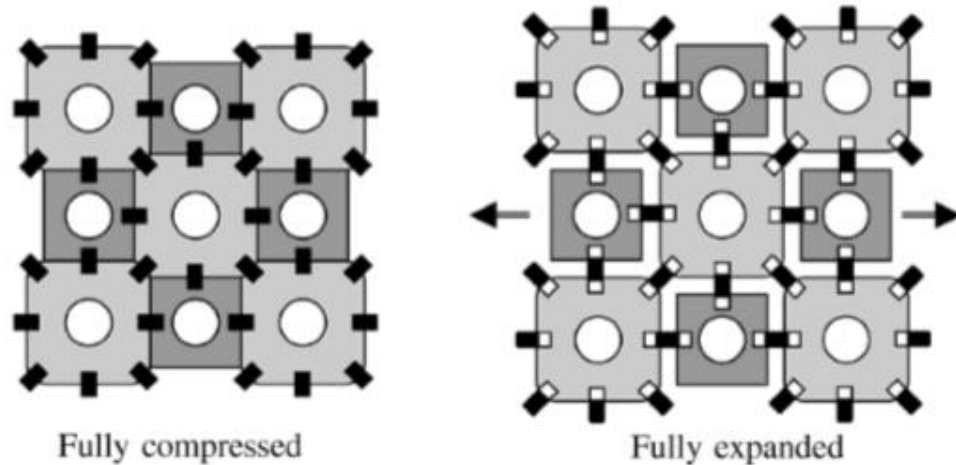


Figure 2. 8: Keyed brick auxetic structure (Evans and Alderson 2000).

2.3. Applications of Auxetic structures

Auxetic structures have been important in biomedical field. Cellular and microporous material have ability to open cavities like dilators. These are used to open small vessels and blocked arteries. Hollow rod of auxetic structure open the vessel by lateral (Evans and Alderson 2000).

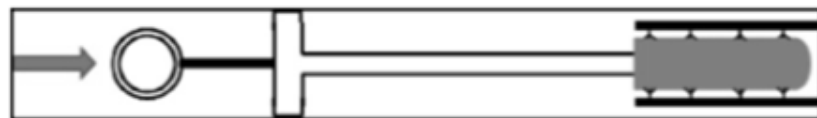


Figure 2. 9: Dilator to open small vessels (Bhullar *et al.*, 2015).

Heart diseases cause the blockage of arteries which are then opened by artery dilator. A mesh of wire forming a cylindrical form is inserted in blocked artery and angioplasty balloon open the stent causing unblocking of artery. Reentry drug eluting stents are introduced to avoid auto immune response and proliferation. Auxetic stents are one of these new vision stents (Bhullar *et al.*, 2015).



Figure 2. 10: Drug delivering auxetic stent (Bhullar *et al.*, 2015).

For sensor and actuator applications functional composite materials are used. Piezoelectric composites are used for medical ultrasound and naval sonar. This composite consists of ceramic rods inside polymer matrix. Recent designs prefer auxetic polymer matrix than conventional polymer matrix (Evans and Alderson 2000).

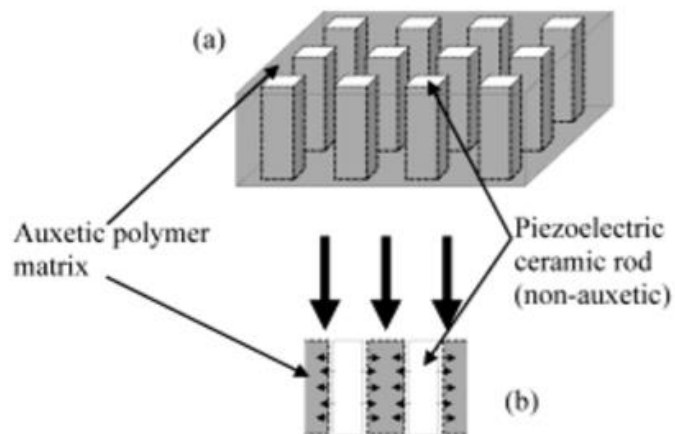


Figure 2. 11: (a) Piezoelectric matrix. (b) Unit of piezoelectric matrix (Evans and Alderson 2000).

Auxetic material has an interesting application which is smart filters and smart bandages. The drug is introduced within the pores of auxetic structure. When stressed is applied the drug is released from the pores. This principle is used for smart bandages. For smart filters the enlargement of pores due to stretching can give filtration. The swelling of wounds can cause stretch in smart bandage releasing anti swelling drug. Release of drug is dependent on the pull done by the swelling (White and Liz 2009).



Figure 2. 12: Smart bandage releasing drug on wound swelling (White and Liz 2009).

For controlled drug delivery wound healing application macro scale auxetic model was prepared. It composed of mechanism of drug release and auxetic film. The mechanism of drug release actually extends the auxetic film. This extension cause opening of auxetic film thus releasing the drug for wound healing. The amount of drug was controlled by actuation mechanism (Mir *et al.*, 2014).

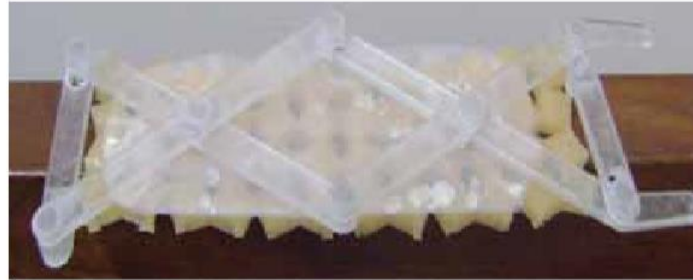


Figure 2. 13: Macro model of smart bandage for controlled drug delivery and porosity (Mir *et al.*, 2017).

CHAPTER 3 MATERIALS AND METHODS

3.1. Materials

3.1.1. Auxetic membrane

Polyurethane (PU) PMC-744 (Smooth-On) polymer was used for the synthesis of auxetic membrane. The hardness of polyurethane was 44 shore-A. It is elastic and biocompatible in nature.

3.1.2. Device Skeleton

For the fabrication of device skeleton biocompatible polyester PLA (poly lactic acid) was used. It has strength and hardness of 92 shore-D. It is thermoplastic.

3.1.3. Hydrogel (self-actuator)

Hydrogel synthesis was done by following chemicals. PVA (Polyvinyl alcohol) of BioChemica with molecular weight of approximately 72000 g/mol and hydrolysis of 85-89% degree was used. Avonchem (UK)'s chitosan was used. From Daejung Chemical Co. (Siheung, Korea) Gelatin (4055–1405) was procured. For chemical crosslinking Glutaraldehyde (Daejung Chemical Co.) was used as a 25% (wt%) aqueous solution. Distilled water and Acetic Acid was used as solvents in process of hydrogel synthesis.

3.1.4. Software

CAD design of auxetic structure and device skeleton was designed on Pro-E wildfire 3.0. For laser cutting Auto CAD software was used while for 3D printing Maker Bot software was used. For expansion measurements MATLAB R2011 was used.

3.2. Methods

3.2.1. Fabrication of auxetic film

Polyurethane was cast in 0.1 mm thickness film. Auxetic 2D rotating square geometry was designed on Pro-E software.

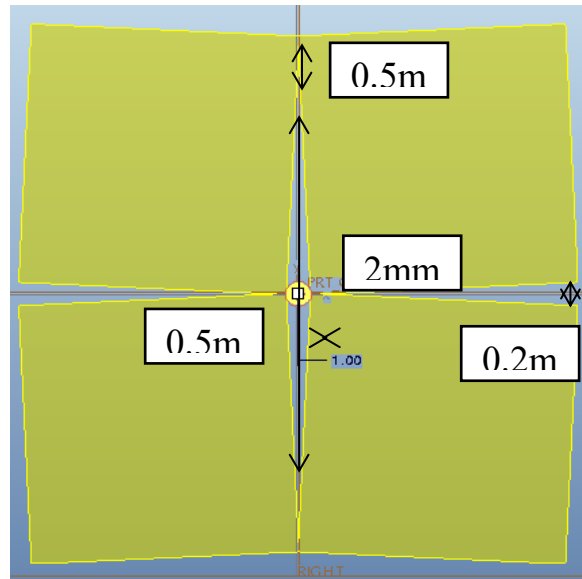


Figure 3. 1: Rotating square auxetic single unit CAD Design.

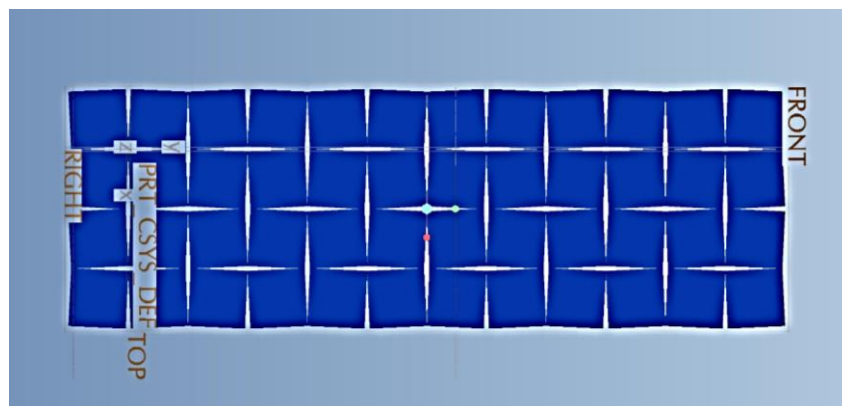


Figure 3. 2: Auxetic film CAD design.

JinQiang 9060 laser cutting machine was used to cut polyurethane film in 10x20 mm size and to fabricate rotating square CAD. The speed of laser was 2 mm/min and power was 7W.

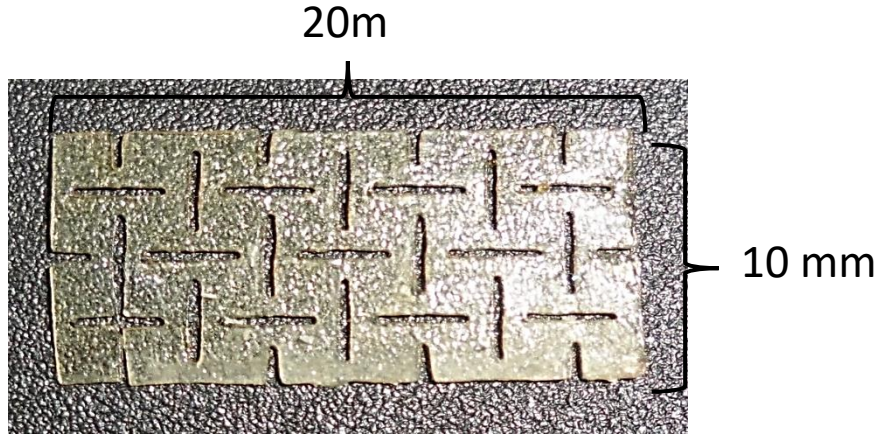


Figure 3. 3: Fabricated auxetic film.

3.2.2. Device skeleton fabrication

Pro-E software was used to design the device skeleton.

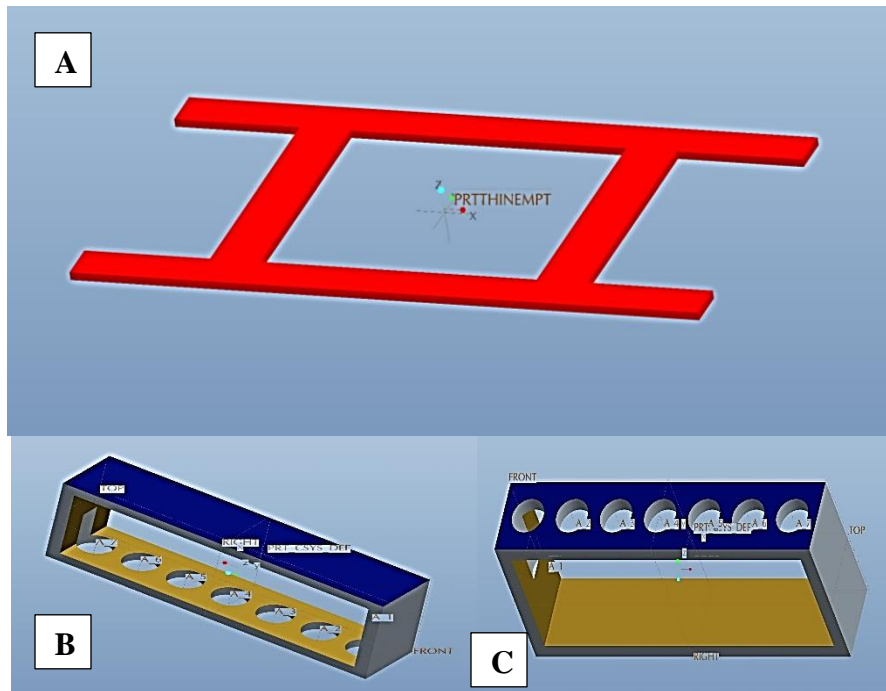


Figure 3. 4: (A) Device skeleton base. (B) Constraining clamp top view. (C) Constraining clamp bottom view.

The CAD design was fabricated by 3D printer. Qidi Tech 2015-2016 3D printer was used to print the device skeleton.

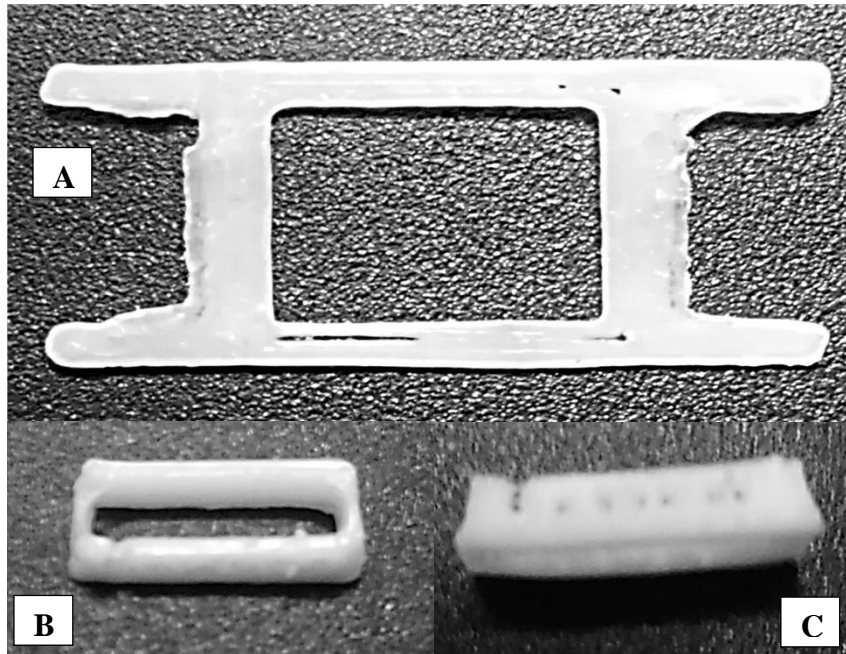


Figure 3. 5: (A) 3D printed PLA device skeleton base. (B) 3D printed constraining clamp top view. (C) 3D printed constraining clamp bottom view.

3.2.3. Hydrogel synthesis

Solution of 5% PVA was made in distilled water. It was stirred on magnetic stirrer at 150C for 2 hours. A 0.5% Chitosan solution of 0.5% was prepared in 1% acetic acid solution with stirring at 30C for 30 minutes. Gelatin was dissolve in distilled water to make 7% solution of it. It was stirred for 1 hour at 50C. Glutaraldehyde 0.063% was used as a crosslinking agent.

PVA, chitosan and gelatin solutions were then mixed along with cross linker. This mixture was then pour into a mold to air dry at room temperature.

3.2.4. Hydrogel compositions

Hydrogel with different ratios were prepared. Completely dried hydrogel was then cut into 5x5x1 mm size to measure its swelling.

Table 3. 1: Different hydrogel compositions.

Sr. No.	Compositions			
	Glutaraldehyde	Chitosan 0.5%	PVA 5%	Gelatin7%
A	0.063%	1	1	3
B	0.063%	1	1	4
C	0.063%	1	2	4
D	0.068%	1	1	4
E	0.068%	1	1	5
F	0.068%	1	1	6
G	0.068%	1	1	7
H	0.068%	1	1	8

Out of eight compositions four compositions were selected for device testing. The criteria for selection was

- Swelling of hydrogel (more than 2.5 mm³)
- Dimensionally stable

- Do not break when constrained

According to above criteria composition E, F, G and H were selected for validation of self-actuating system.

3.2.5. Fabrication of self-actuating device

Hydrogel was attached at sides of PLA device skeleton. Polyurethane auxetic film was bonded with hydrogel piece. Hydrogel was constrained along two axis by 3D printed clamps.

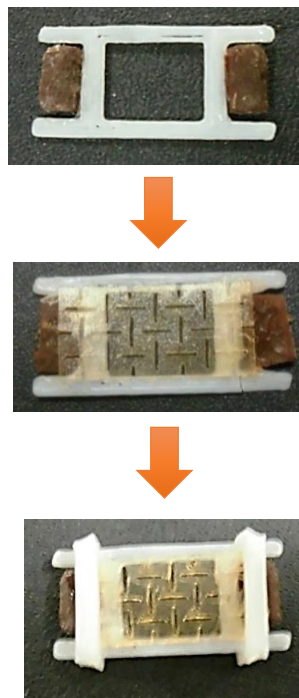


Figure 3. 6: Fabrication of self-actuated auxetic device.

3.2.5. Validation of the self-actuating system

Hydrogel was introduced to water. Expansion of auxetic film was observed.

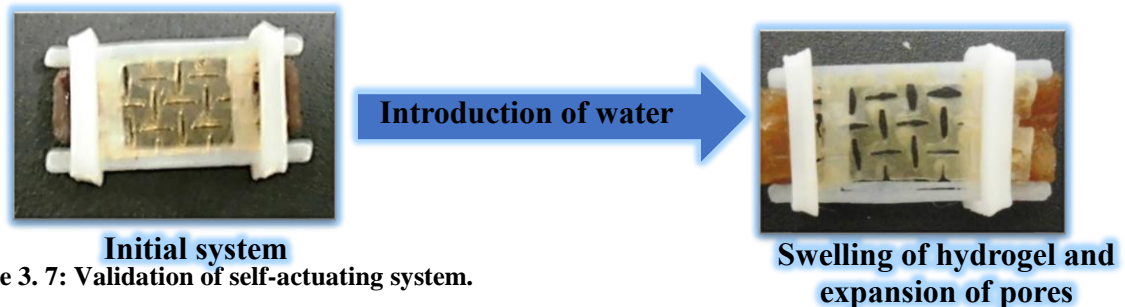


Figure 3. 7: Validation of self-actuating system.

Selected hydrogel compositions were tested. Expansion of auxetic film was observed. Images were taken to measure the expansion.

3.2.6. Auxetic film expansion analysis

The measurement of auxetic film expansion was done by image acquisition method and calculation through MATLAB R2011 image tool.

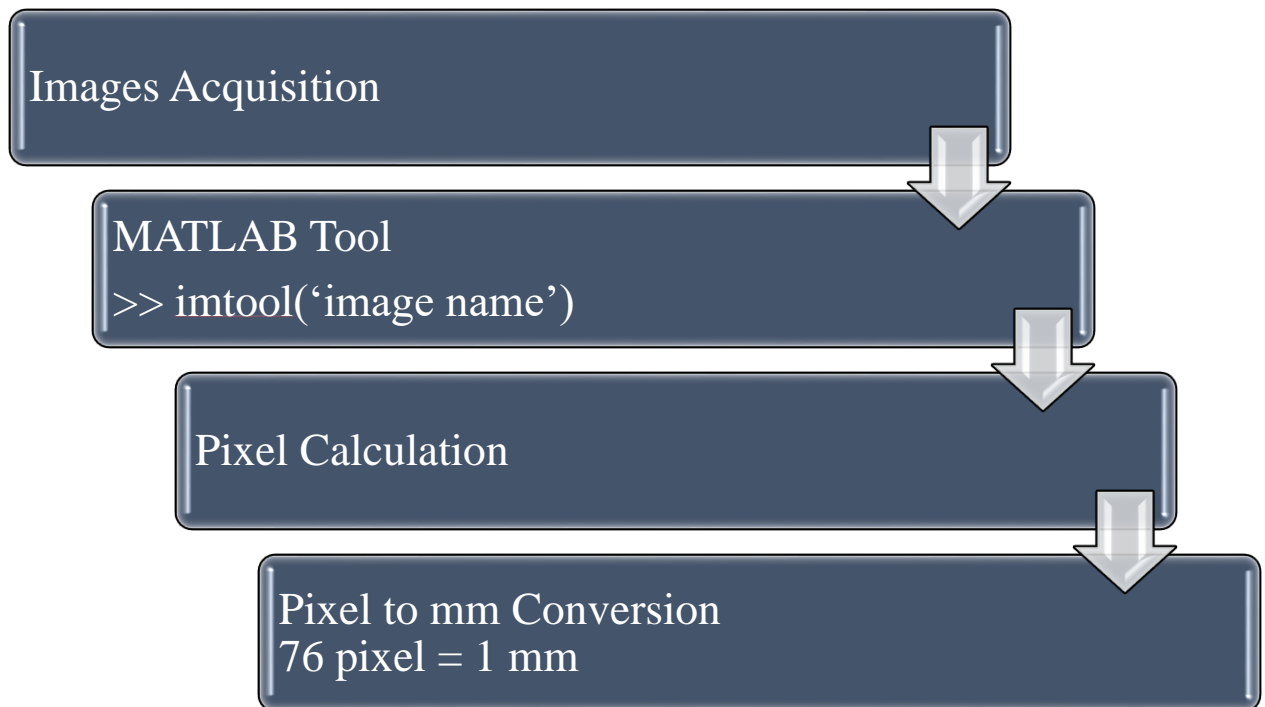


Figure 3. 8: Flow chart of auxetic film expansion analysis.

3.2.6.1. Image acquisition

Images of auxetic film expansion were taken from Casio EXILIM 20.1 MP camera. The size of images was 5152x3864 pixels.

3.2.6.2. MATLAB tool

For Image Processing MATLAB image processing tool was used accessed with following code.

```
>> imtool('image name')
```

3.2.6.3 Pixel measurement and calculation

Image of a piece of a known length 10x10 mm was taken and was compute the calibration scale factor in mm per pixel.

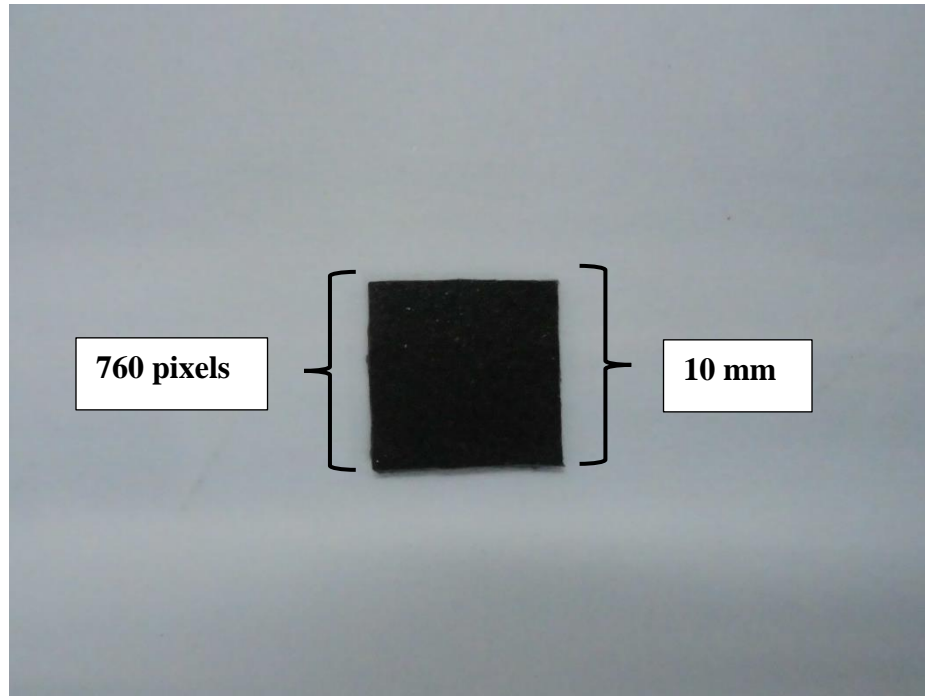


Figure 3. 9: Known length piece for scale.

According to the MATLAB image tool

760 pixels = 10 mm

76 pixels = 1 mm = 1000 um

3.2.7. Mechanical testing

Mechanical testing was done by SHIMADZU Load cell model ML2960. The graphs and data was taken from the load cell autograph software.

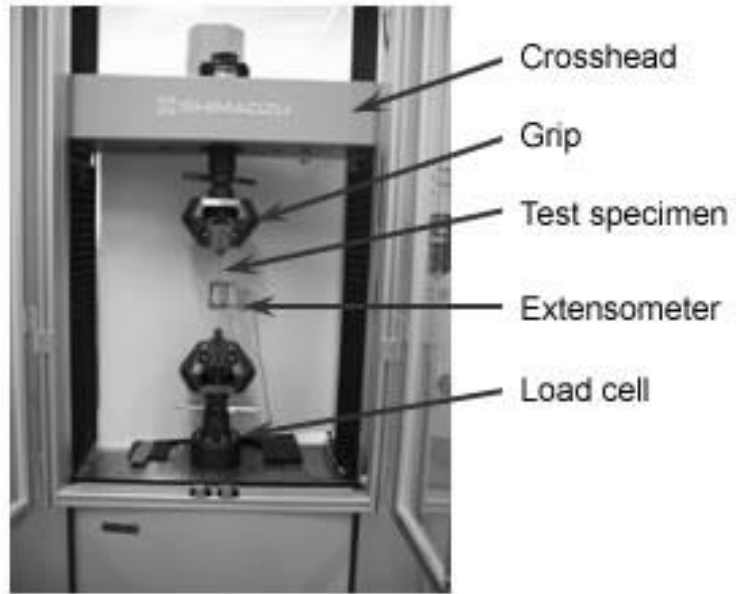


Figure 3. 10: Mechanical testing machine.

Mechanical testing was done on the rate of 2 mm per minute.

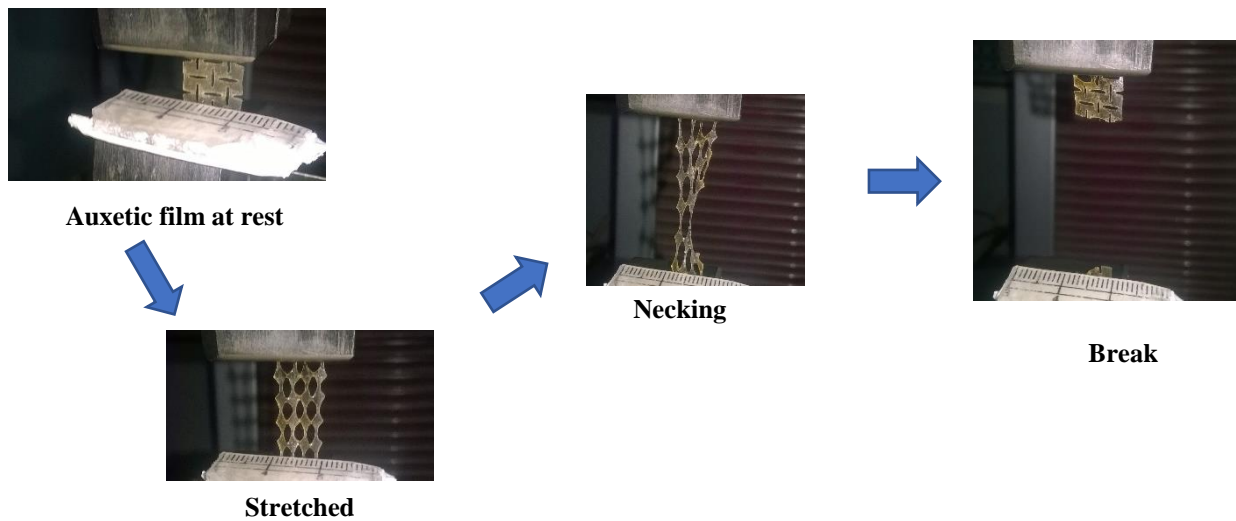


Figure 3. 11: The process of mechanical testing.

CHAPTER 4 RESULTS AND DISCUSSION

4.1. Hydrogel compositions expansions

Hydrogel was introduced to water and its size was measured after every 30 minutes.

Table 4. 1: Different hydrogel compositions and their swelling behavior.

Sr. No.	Composition				Hydrogel swelling with time (mm ³)					
	Glutaraldehyde	Chitosan 0.5%	PVA 5%	Gelatin 7%	30 mins	60 mins	90 mins	120 mins	150 mins	180 mins
A	0.063%	1	1	3	0.7	1	1.6	2	2.5	2.5
B	0.063%	1	1	4	1.2	1.8	2.3	2.7	3	3.5
C	0.063%	1	2	4	0.3	0.7	1.1	1.4	1.6	1.6
D	0.068%	1	1	4	0.6	1.1	1.7	2.1	2.5	2.5
E	0.068%	1	1	5	0.8	1.4	1.8	2	2.6	2.9
F	0.068%	1	1	6	0.8	1.8	2.2	2.6	3.3	3.4
G	0.068%	1	1	7	0.9	1.9	2.5	3.1	3.5	3.7
H	0.068%	1	1	8	1	2.1	2.8	3.3	3.7	4

Composition A was taken from the literature but its expansion was not enough and did not expand the pores of auxetic film. The composition B had good expansion but it was soft and broke easily.

Composition C and D had less expansion. Composition E, F, G and H were selected to test self-actuated system. The expansion rate of hydrogel can be shown in graph form.

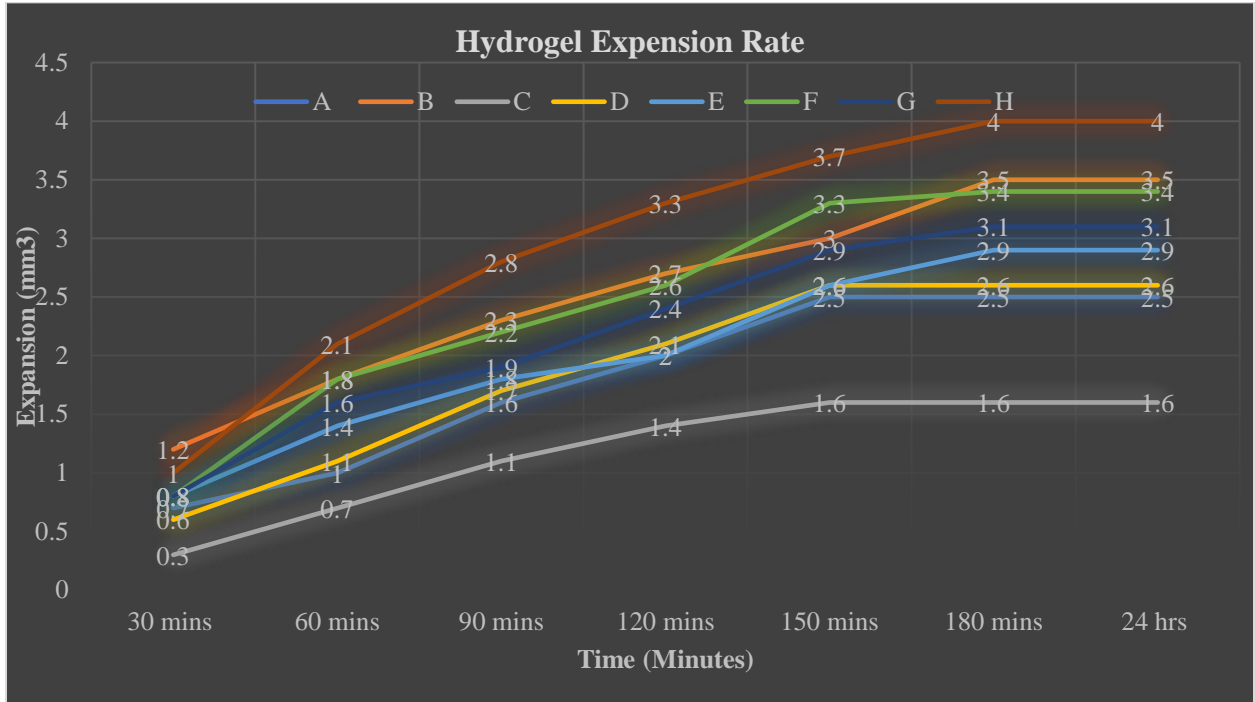


Figure 4. 1: Hydrogel expansion graph.

4.2. Measurement of auxetic film expansion

The expansion of auxetic film was measured by image processing through MATLAB. The expansion of pores in micrometers is shown in the table below.

Table 4. 2: Expansion measurement of the membrane

Name of composition	Initial measurement (um)	Final measurement (um)	Total expansion (um)
A	395	618	223
E	395	1224	829
F	395	1342	947
G	395	1553	1158
H	395	1895	1500

The table values show that composition H can expand pores of auxetic film up to 1500 um which is highest of the all compositions. We can use any of the composition according to our pore expansion desire.

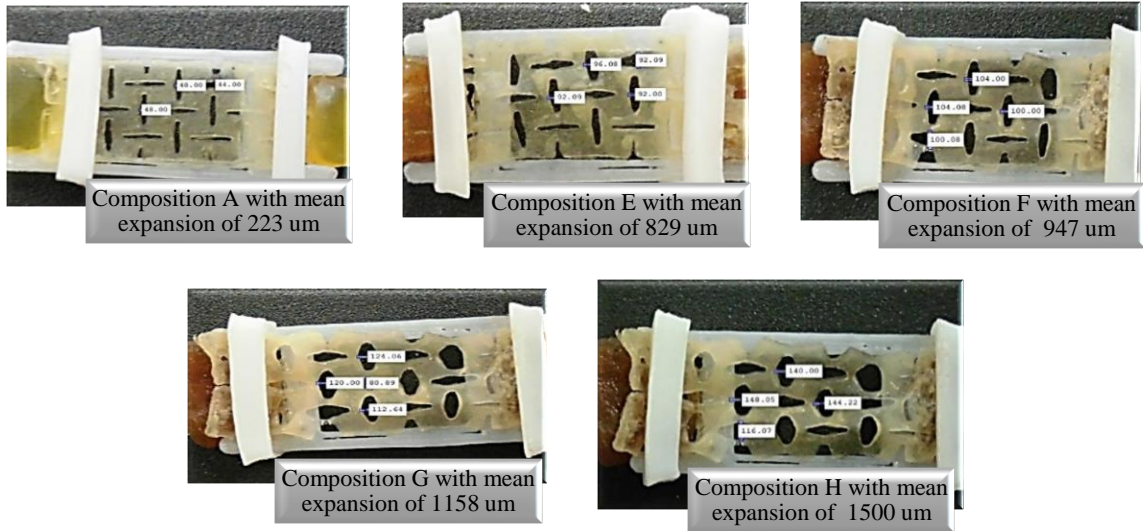


Figure 4. 2: Images of self-actuated auxetic system with open pores.

4.3. Mechanical testing

Mechanical testing gives two graphs. One stress vs strain graph other force vs displacement graph.

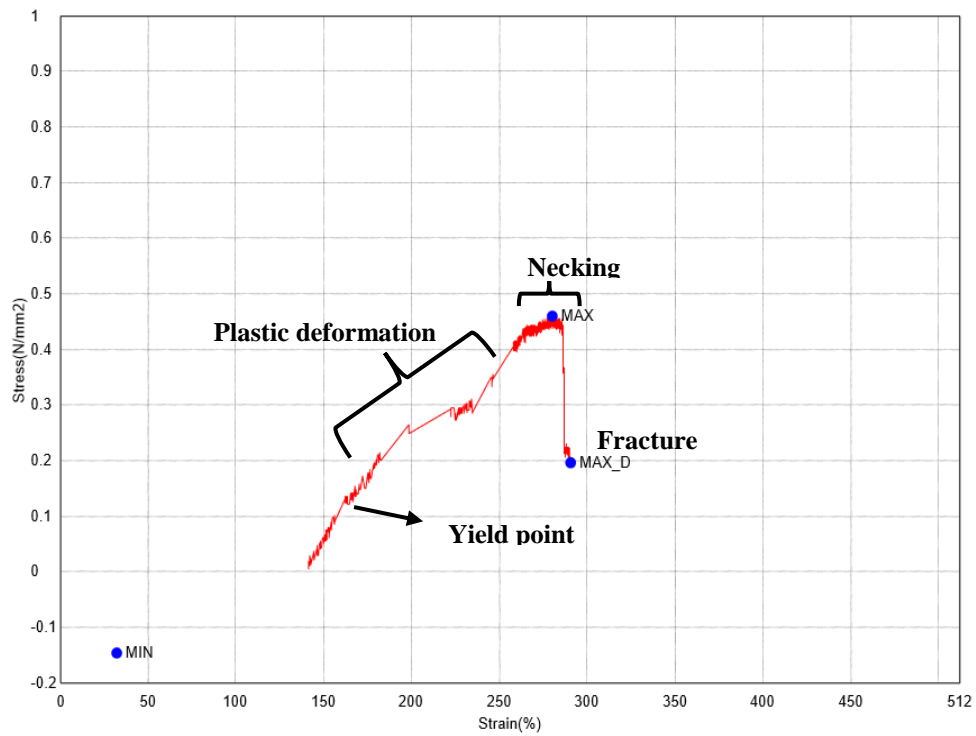


Figure 4. 3: Stress-Strain graph of mechanical testing

Stress vs strain graph showed the yield point to be at approximately at 0.15 mpa stress. Till this point the auxetic membrane showed elastic deformation. The region of plastic deformation was greater than elastic deformation. The graph showed the membrane was elastic and can stretched upto approximately 250% of its original length.

Maximum strength was 0.4 mpa and maximum strain wan 256%. Elastic modulus was calculated through the formula.

Elastic modulus = Stress / Strain.

Calculated elastic modulus was 0.0015625 mpa. This value showed the auxetic membrane was very much elastic.

Table 4. 3: Conventional polymers and their elastic modulus.

Sr. No.	Polymer	Elastic modulus
1	Teflon (PTFE)	0.4 - 0.55
2	Silicon elastomer	0.005 – 0.02
3	Natural rubber	0.0015 – 0.025
4	Nylon (PA)	2.62 – 3.2
5	Polypropylene (PP)	0.896 – 1.55
6	Neoprene	0.0007 – 0.002

Compared to other conventional polymer’s elastic modulus the auxetic membrane was more flexible and can bear load.

Another graph of force vs displacement was also obtained.

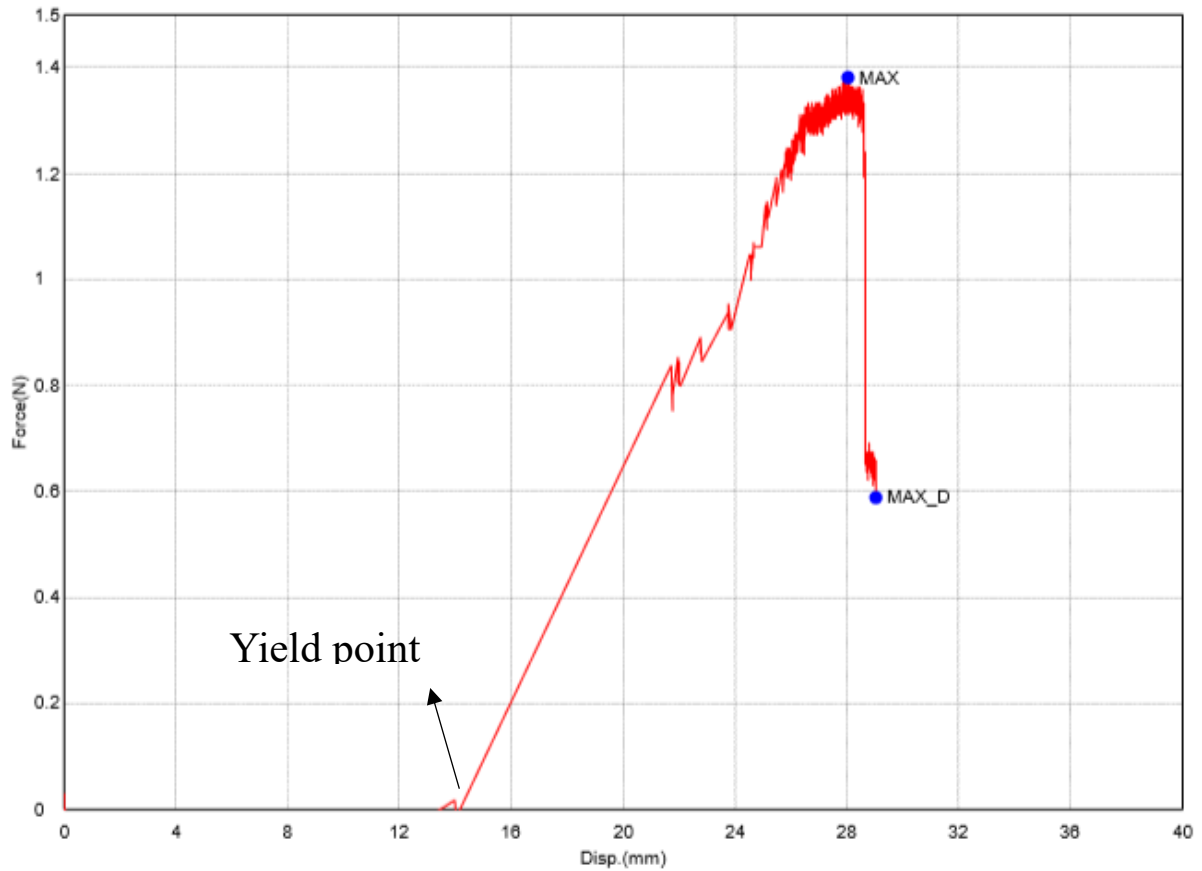


Figure 4. 4: Force-Displacement graph of mechanical testing

Force vs displacement graph showed maximum displacement of 24.8 mm and maximum force applied before membrane breakage was 1.4 N. Poisson's ratio was calculated from these obtained values from formula.

$$\text{Poisson's ratio} = \text{Lateral Strain} / \text{Axial Strain}$$

This gave Poisson's ratio value of -0.31 . the membrane was auxetic so its Poisson's ratio should be negative.

CHAPTER 5 CONCLUSION AND FUTURE PERSPECTIVES

5.1. Conclusion

In this project, self-actuated auxetic structure has been developed. The hypothetical design was fabricated and validated in research lab. Hydrogels of different compositions were used to control the porosity of the auxetic membrane. Mechanical testing of the auxetic membrane was done. This type of self-actuated mechanism can be utilized in variety of biomedical applications like smart bandage for wound healing, stent expansion etc. The self-actuated auxetic system is micro scale and can be used in micro scale applications.

5.2. Future prospective

In the fields of science and medicine, auxetic structure are making their way in devices due to their properties. they show strength and flexibility at the same time. The micro scale design fabricated in this research can be used as smart bandage. Healing drug can be introduced in the slits of the auxetic structure which can release later by swelling of hydrogel acting as actuator and releasing the medicine from the pores.

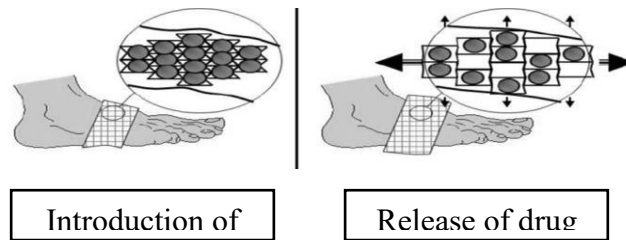


Figure 5.1: Drug releasing smart bandage.

CHAPTER 6 REFERENCES

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