Characterization of Biodegradable Polymer Subjected to Different Solvent Mixture

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Abstract - The objective of this research was to determine the optimum solvent mixture to improve flexibility of PLA solvent cast film. In spite of trials of the reducing polymeric waste and strong limitations concerning storage and product end-life cycle show, the amount of the non-degradable polymer slowly has become ballast for the environment. PLA is a alternative biodegradable polymer for food packaging industry. PLA films were formed from mixed solvent solutions (chloroform & Dichloromethane) and Single solvent solutions, i.e., chloroform & Dichloromethane, using solvent casting technique. The ratio of solvents used for preparing a specimens were 25ml of chloroform, 30ml of chloroform & 30ml of Methylene dichloride(with & without PEG), 50ml of chloroform & 50ml of Dichloromethane. To know about the optimum solvent solutions, tensile test and Hardness tests were conducted. Tensile test shows the variation of stress-strain curve over 5 different specimens, out of which a specimen with 3gms of PLA dissolved in 50ml of dichloromethane exhibited a good flexibility. Hardness test shows that, hardness number is less for a specimen with 30ml of dichloromethane which exhibited good ductility of a material compared to other specimens.

Keywords- Poly lactic acid, biodegradable polymer, chloroform, dichloromethane,

INTRODUCTION

Growing impact of the plastic goods on the growth of human life standard is connected with a continuing expansion of petroleum-based polymers. In spite of trials of the reducing polymeric waste and strong limitations concerning storage and product end-life cycle show, the amount of the non-degradable polymer slowly has become ballast for the environment. Therefore, the attempt of introducing biodegradable polymers in industrial-scale production gained ground among scientists. The area of biodegradable material application is continuously extending due to their improving properties which in many cases resemble petrochemical polymers. In spite of many studies concerning the usage of biodegradable polymers, such as poly (lacticacid) (PLA), poly (butylenes adipate-co-terephthalate)(PBAT) polypropylene carbonate (PPC) , and starch , their commercial application is still not very common. Packaging industry appears as a branch which due to relatively low opportunity towards mechanical properties allows for wide application of fully biodegradable polymers on a bigger scale. The small thermo-mechanical steadiness of green composites, next to comparatively elevated price, became their biggest drawback in comparison to petroleum-based non-biodegradable polymers. These polymers forced the researchers to make use of recycled thermoplastic eco-friendly polymers as a matrix for composites filled with organic and inorganic fillers. To reduce the rate of contamination is by developed article from biodegradable material. Eco-friendly material are those which can be degraded in the existence of ecological condition like soil, moisture, microorganism, light, heat, etc. and end products of this is not dangerous to environment. Lactic acid has received attention for use in wide range of application typically as it acts as a monomer for the production of biodegradable poly (lactic acid) or polylactide (PLA).PLA can be formed chemically and biotechnologically but biotechnological routes are mostly preferred because of ecological concerns and inadequate nature of petrochemical feedbacks. Global efforts have been made for the production of lactic acid and PLA with good quality yield and low cost management. For the pilot scale production of lactic acid though biotechnological route3, there have been various requirement for high productivity i.e. cheap raw material, lactic acid producing microorganism, fermentation approach, type of bioreactor and finally purification of optically pure lactic acid for production of highly crystalline lactic acid. Polylactic acid (PLA) is most acceptable material for biomedical applications due to biocompatible nature and tissue integration. The glass transition temperature is near to body temperature, and hence, it is brittle in nature. In addition, it has sufficient mechanical strength but due to its low fracture stiffness and high biodegradability compelled to blend it with some other polymers. PCL is another potential polymer for the application. It is hydrophobic, semi-crystalline polymer with high solubility, low melting point (59–64°C), and exceptional blend compatibility. Due to low mechanical loading capability and slow degradation time, it can be tailor made by mixing with other biomaterials as per requirements. In this research work, a co polymeric scaffold was fabricated using PCL and PLA to increase strength and controlled hydrolytic degradation. Also, a non proliferative agent was coated onto the scaffold by spray coating method, and a sustained drug release profile was observed.

MATERIAL & METHODS

The specimens were prepared in laboratory by solvent casting method. 3 or 4gms of PLA was dissolved in beaker containing 25-50ml of Chloroform or dichloromethane and stirred well. As soon as the solution becomes clear, 0.4-0.5grms of PEG was added in that solution. Solutions were stirred until it becomes apparent and normalize properly. The solution was casted in glass Petri dish of uniform size 20×15 cm and dried at room temperature. The film was cautiously detached from the die with the aid of spatula.

Figure 1: Chloroform, Dichloromethane & Polyethylene glycol
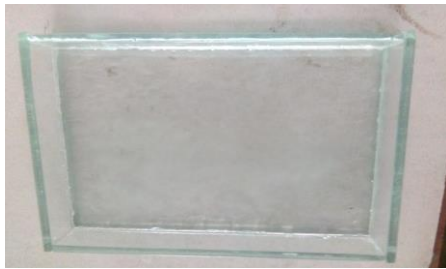

Figure 2: prepared Die

The PLA Film was prepared in the laboratory using solvent casting method. The PLA film of same concentration i.e. 4grms was added with 30 ml of Chloroform, dichloromethane. PEG was selected with different concentration i.e. 0.3grms, 0.4grms. fig.3 shows 5 film samples that prepared with 5 different compositions as per the compositions mentioned in the table 1.

Figure 3: PLA Specimens with different combination

Specimens Preparation:

1. *PLA film 1*
   - PLA (3gms) & poly ethylene glycol (PEG400-0.5gms) were dissolved in 25ml of solvent of chloroform.
   - The solution was stirred for 2 hours at room temperature.
   - Cast films were dried at room temperature & then peeled from the glass plate.

Figure 4: PLA specimen with only chloroform (0.5gms PEG)

2. *PLA film 2*
   - PLA (4gms) & poly ethylene glycol (PEG400-0.4gms) were dissolved in 60ml of solvents i.e. (dichloromethane 30ml & chloroform 30ml).
   - The solution was stirred for 2 hours at room temperature.
   - Cast films were dried at room temperature & then peeled from the glass plate.

Figure 5: PLA specimen with chloroform & dichloromethane (PEG 0.4gms)

3. *PLA film 3*
   - PLA (4gms) were dissolved in 60ml of solvents i.e. (Methylene chloride 30ml & chloroform 30ml).
   - This film is prepared without using plasticiser i.e. PEG
   - The solution was stirred for 2 hours at room temperature.
   - Cast films were dried at room temperature & then peeled from the glass plate

Figure 6: PLA specimen with chloroform & dichloromethane (without PEG)

4. *PLA film 4:*
- PLA (3gms) were dissolved in 50ml of chloroform.
- This film is prepared without using plasticiser i.e. PEG
- The solution was stirred for 2 hours at room temperature.
- Cast films were dried at room temperature & then peeled from the glass plate

Figure 7: PL[A] ...asticizer)

5. *PLA film 5:*
- PLA (3gms) were dissolved in 50ml of dichloromethane
- This film is prepared without using plasticiser i.e. PEG
- The solution was stirred for 2 hours at room temperature.
- Cast films were dried at room temperature & then peeled from the glass plate.

Figure 8: PLA Specimen with only dichloromethane (without Plasticizer)

Table 1: Composition for Different PLA specimens

SL NO	PLA(gms)	Chloroform(ml)	Methylene dichloride(ml)	PEG (gms)
1	3	25	-	0.5
2	4	30	30	0.4
3	4	30	30	
4	3	50	-	
5	3	-	50	

EXPERIMENTATION

1. *Tensile test*

Tensile testing, also recognized as tension testing, is a basic materials science and engineering test in which a sample is subjected to a pulling force until breakdown. Properties that that could be obtained through a tensile test are: maximum tensile strength, breaking strength, percentage elongation and decrease in area. From the collected data, Young's modulus, Poisson's ratio, yield strength, and strain-hardening characteristics were analysed. Uniaxial tensile testing is normally used for obtaining the mechanical uniqueness of isotropic materials.

Figure 9: Tensile testing machine

Tensile testing is most frequently carried out at a material testing laboratory. The ASTM D638 measures plastics tensile properties including maximum tensile strength, yield strength, elongation and Poisson's ratio. The most frequent testing machine used is the universal testing machine. There are two types: hydraulic powered and electromagnetically powered machines. The machine should have the suitable capability for the test specimen being tested. There are four major parameters: force capacity, speed, precision and accuracy. Force ability refers to the fact that the machine should be capable to produce sufficient force to break the specimen. The machine should be able to apply the force speedily or gradually sufficient to appropriately imitate the actual function. lastly, the machine should be capable of precisely and exactly measure the gauge length and forces applied; for example, a large machine that is intended to measure long elongations may not work with a brittle material that experience short elongations earlier to fracturing. Arrangement of the test specimen in the testing machine is decisive, because if the specimen is misaligned, either at an angle or offset to one side, the machine will put forth a bending force on the specimen. This is in particularly bad for brittle materials, because it will severely skew the results. These circumstances can be minimized by means of sphere-shaped seats or U-joints among the grips and the test machine. If the first portion of the stress–strain curve is curved and not linear, it shows, the specimen is uneven in the testing machine.

2. *HARDNESS TEST*

Hardness is a ability of a material to resist indentations or surface abrasion. Some materials (e.g. metals) are harder than others (e.g. plastics). Macroscopic hardness is usually

characterized by well-built intermolecular bonds, however the activities of solid materials beneath force is complex; therefore, there are different measurements of hardness: scratch hardness, indentation hardness, and rebound hardness.

- **Scratch Hardness:** it is the measure of how resistant a sample is to fracture or permanent plastic deformation due to friction from a sharp object. The principle is that an object made of a harder material will scratch an object made of a softer material. When testing coatings, scratch hardness refers to the force necessary to cut through the film to the substrate. The most familiar test is Mohs scale, which is used in mineralogy. One tool to make this measurement is the sclerometer. One more tool used to make these tests is the pocket hardness tester. This tool consists of a scale arm with graduated markings attached to a four-wheeled carriage. A scratch tool with a sharp rim is mounted at a fixed angle to the testing surface. In order to use it a weight of known mass is added to the scale arm at one of the graduated markings, the tool is then drawn across the test surface. The use of the weight and markings allows a known pressure to be applied without the need for complicated machinery

- **Indentation Hardness:** Indentation hardness measures the resistance of a sample to material deformation due to a constant compression load from a sharp object; they are primarily used in engineering and metallurgy fields. The tests work on the basic principle of measuring the significant dimensions of an indentation left by a purposely dimensioned and loaded indenter. Rockwell, Vickers, Shore, and Brinell are the commonly used hardness tests..

- **Rebound Hardness:** Rebound hardness, also known as dynamic hardness, measures the height of the "bounce" of a diamond-tipped hammer dropped from a fixed height onto a material. This type of hardness is related to elasticity. The mechanism which is used to have this measurement is known as a scleroscope. Leeb rebound hardness test and Bennett hardness scale are the two scales that measures the rebound hardness

RESULT & DISCUSSION

1. *Tensile Test*

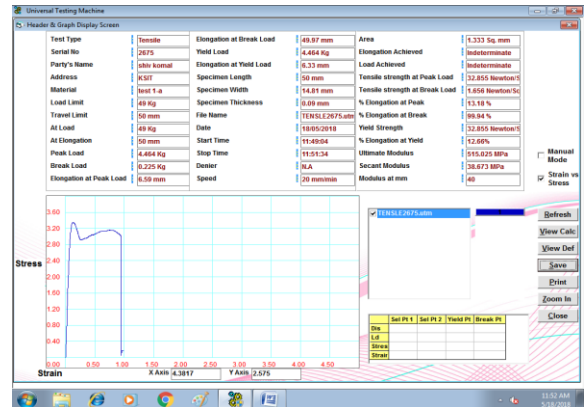*i.* *PLA (3gms) & poly ethylene glycol (PEG400-0.5gms) were dissolved in 50ml of chloroform.*

Figure 10: Stress-Strain curve for PLA specimen with chloroform (with 0.4gms of PEG)

The first iteration of the tensile strength for PLA composites is shown in Figure 10. The highest values of tensile strength was recorded at 33.345Mpa (i.e. shown in table 2) for PLA (3gms) & chloroform (50ml) with poly ethylene glycol (PEG400-0.5gms).

ii. *PLA (4gms) & poly ethylene glycol (PEG400-0.4gms) were dissolved in 60ml of solvents i.e., (Methylene chloride 30ml & chloroform 30ml)*
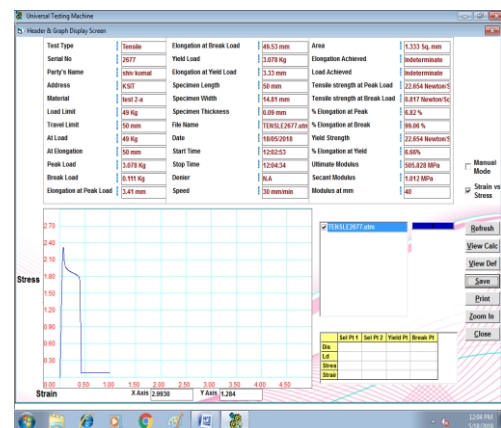

Figure 11: Stress-Strain curve for PLA specimen with chloroform & dichloromethane (with 0.5gms of PEG)

The second iteration of the tensile strength for PLA composites is shown in Figure 11. The highest values of tensile strength was recorded at 23.544Mpa (i.e. shown in table 2) for PLA (4gms) & 60ml of solvents i.e., (Dichloromethane 30ml & chloroform 30ml) with poly ethylene glycol (PEG400-0.4gms).

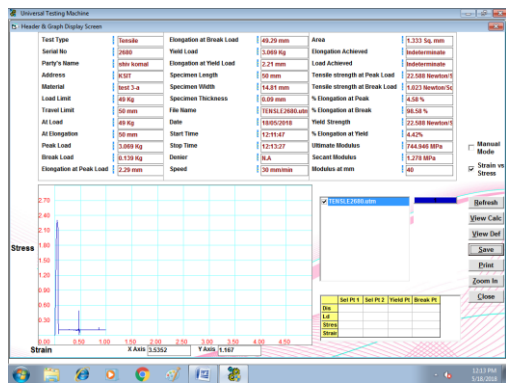iii. PLA (4gms) were dissolved in 60ml of solvents (Methylene chloride 30ml & chloroform 30ml)

Figure 12: Stress-Strain curve for PLA specimen with 30ml chloroform and 30ml dichloromethane (without PEG)

The third iteration of the tensile strength for PLA composites is shown in Figure 12. The highest value of tensile strength was recorded at 22.563Mpa (i.e. shown in table 2) for PLA (4gms) which was dissolved in 60ml of solvents i.e., (Dichloromethane 30ml & chloroform 30ml) without the use of poly ethylene glycol (PEG400).

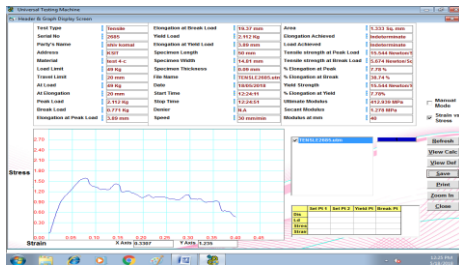iv. PLA (3gms) were dissolved in 50ml of solvent (chloroform)

Figure 13: Stress-Strain curve for PLA specimen with 50ml chloroform (without PEG)

The fourth iteration of the tensile strength for PLA composites is shown in Figure 13. The highest values of tensile strength was recorded at 15.969Mpa (i.e. shown in table 2) for PLA (3gms) which was dissolved in 50ml of solvent i.e, chloroform.

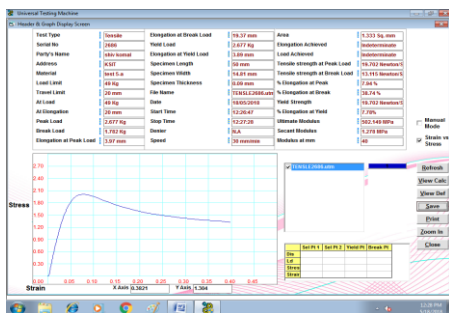v. PLA (3gms) were dissolved in 50ml of Dichloromethane.

Figure 14: Stress-Strain curve for PLA specimen with 50ml dichloromethane (without PEG)

The fifth iteration of the tensile strength for PLA composites is shown in Figure 14. The highest values of tensile strength was recorded at 19.677Mpa (i.e. shown in table 2) for PLA (3gms) which was dissolved in 50ml of dichloromethane.

Table 2: results of Tensile Test

Specimen No.	Actual width (mm)	Actual thickness(mm)	Ultimate stress (Mpa)	Elongation (%)	Modulus of Elasticity (Gpa)
1-a	14.81	.091	33.354	9.8	3.403
1-b	14.81	.094	46.107	2.4	19.211
1-c	14.81	.093	39.24	2.4	16.35
2-a	14.81	.088	23.544	4.4	5.3509
2-b	14.81	.091	26.487	1.4	18.92
2-c	14.81	.090	26.487	0.8	33.1087
3-a	14.81	.097	22.563	0.8	28.2037
3-b	14.81	.100	15.696	0.6	26.16
3-c	14.81	.099	13.734	0.7	19.62
4-a	14.81	.102	25.506	1	25.506
4-b	14.81	.105	12.753	1	12.753
4-c	14.81	.103	15.969	4	3.99225
5-a	14.81	.094	19.677	4	4.91925
5-b	14.81	.096	16.677	2.4	6.9487
5-c	14.81	.097	19.62	0.8	24.525

2. *Hardness Test*

Hardness test was conducted for the PLA specimens mentioned in the table 1 and the results were tabulated below.

Table 3: Hardness test results

Specimen	Hardness number
1	55
2	60
3	62
4	58
5	48

From the above table 3, we can conclude that PLA specimen 3 which was prepared using both the solvents (chloroform & Dichloromethane) have highest hardness number and PLA specimen 5 prepared using only dichloromethane has lowest hardness number.

CONCLUSION

The effects of PLA added into different chemical solutions on the mechanical properties of the composite were studied. From the study, the following conclusions can be concluding:

- From the analysis, it can be concluded that the film with 3gms of PLA & 50ml of Dichloromethane, without adding PEG exhibited a good flexibility.
- Stress strain curve shows that the high crystalline films were brittle while less crystalline films were ductile.
- From the Hardness test, it is concluded that the specimen with good flexibility has a less hardness number compared to other specimens.
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