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'Synthesis of Edible Packaging from Milk Casein.'

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ABSTRACT

Plastics are non-biodegradable and this can be a big threat to environment in this present world. Edible package can be a solution to these environmental problems as they are environment friendly. Protein edible packaging is made using casein protein concentrate as raw material. Casein proteins constitute ~80% of the total proteins in milk. Casein has great potential for producing protein-based edible films. Casein films make food products prone to oxidation. Protein, plasticizers concentration and temperature are studied. The study shows that the increase in protein increases the mechanical properties of the film. Various mechanical and physical properties of the film are also studied. The extensibility of the film was increased in the temperature of 35^0 Celsius. The time required for the film formation was 20hrs. Gelatin is used as the thickening agent allowing it to resist moisture and temperature. Sorbitol is used as a plasticizer, make the protein film softer and control the protein-protein interaction. The objectives of the project are to synthesis edible package from milk casein to study the properties of the film and to determine the applications of these casein edible films in food industry.

KEYWORDS

- **EDIBLE PACKAGE**
- **CASEIN FILMS**
- **TENSILE STRENGTH**
- **THICKNESS OF FILM**
- **HARDNESS TEST**
- **GELATIN**

- **WATER VAPOR TRANSMISSION RATE**
- **APPLICATION IN FOOD INDUSTRY**

CHAPTER 1

INTRODUCTION

Food packaging is packaging for food. A package provides protection, tampering resistance, and special physical, chemical, or biological needs. They are of two types edible and non-edible, where former are thin layers of edible materials which can be applied directly to the surfaces of food products by dipping, spraying or panning.

1.1 EDIBLE PACKAGING

Edible food packaging is basically package wrapped in food. It can be used instead of plastic and paper packages. Edible films can be formed as food coatings and free-standing films, and have the potential to be used with food as gas aroma barrier (Kester and Fennema, 1986). Edible packages are environmentally friendly, bio degradable and recyclable.

1.2 CASEIN

Casein proteins constitute ~80% of the total proteins in milk (Ginger, M.R et al.,). Available in many different by-products from the dairy industry, including calcium and sodium caseinates (CaCas and NaCas. Casein form films readily due to its random coil nature and also has the ability to form intermolecular hydrogen and electrostatic bonds. The films made of proteins are usually brittle and stiff due to strong protein- protein and protein-water interaction (Sung-Woo Cho et al.,)

1.3 CASEIN EDIBLE PACKAGE

Food packaging, made from the milk protein casein, is sustainable, biodegradable, safe and even edible. When used in packaging, they could prevent food waste during distribution along the food chain.

Being edible, the casein packaging will still need to be wrapped in protective outer layers to keep it dry and clean, so while it might not completely replace plastics, it could significantly cut down on the amount of usage. (Dangaran, K.L et al.,).

1.4 OBJECTIVE

1. To synthesis edible package from milk casein.
2. To study the properties of the film.
3. To determine the applications of these casein edible films in food industry.

CHAPTER 2

MATERIALS AND METHODS

3.1 MATERIALS USED

- Sorbitol which makes the protein film softer and control the protein-protein interaction, resulting in increased protein flexibility and there are also some drawbacks on the film properties, they are decrease in the tensile strength and decreased ability to act as a barrier towards moisture, oxygen, aroma and oil.
- Gelatin added more structure to the film, allowing it to resist humidity and high temperatures better.
- Sodium Hydroxide is used to dissolve casein.
- Other materials used for edible packaging is starch and also whey protein.

3.2 OTHER APPARATUS USED

- Beaker
- Burner
- Centrifuge Machine
- UV Visible Spectrophotometer
- Weight Machine
- Test Tubes
- Hot Air Oven

3.2 STANDARD PROCEDURE

30 ml of 0.5mM NaOH and 10 ml of water is taken in a beaker and boil it in a burner.

↓

Take casein and add little by little to the beaker and mix properly for 5 minutes

↓

Then add gelatin and mix well for 5 minutes

↓

Take beaker out of the flame and add sorbitol and mix well for 10 minutes

↓

Pour it to a petri plate and keep it in oven at a temperature of 35̊c for 20 hours.

Flowchart 3.2.1: Synthesis of Edible Casein Films

3.3 FLOWCHART DESCRIPTION

A clean beaker is taken and to which 30 ml of 0.05Mm NaOH is added. Then 10 ml of water is added to the beaker. The casein, sorbitol and gelatin are weighed and then casein is added to the solution little by little and mix well for 5 minutes. Now gelatin is added and also mixed properly for five minutes. Then take the beaker out of the flame and add sorbitol and mix well for 10 minutes. Now take a clean Petri plate and pour the solution to it. Keep it in oven at 35^{o}c for 20 hours.

3.4 OBSERVATIONS:

- When the level of gelatin is reduced and sorbitol is increased, the film formed was not peelable and the texture was also not good.
- When the level of sorbitol is reduced and of gelatin is increased, there was proper film formation and the texture was also good and the film formed was peelable.
- NaOH solution level is decreased from 40ml to 30ml.

3.5 OPTIMIZATION OF TEMPERATURE AND TIME:

The time and temperature is optimized by considering the rate of drying, its peelable properties and thickness.

At 45 ̊c the film formed was brittle and was not able to peel off

At 40 ̊c the time taken for drying was more and the thickness need to be more in order to peel it off from the petri plate.

At 35 ̊c the time taken for film formation was less and even though the thickness is less it was peelable.

The optimized time is 20hrs and if the time is or the film becomes hard or sometimes brittle and thus make it difficult to peel.

3.6 APPLICATIONS DONE USING STANDARDIZED EDIBLE PACKAGE

1. To determine the browning reaction of fresh cut apples.
2. To Synthesis 3 in 1 coffee sachets made from film.
3. To differentiate the sogginess of corn flakes with and without packaging material.

3.7 MECHANICAL PROPERTIES OF FILM

3.7.1 TENSILE STRENGTH

Tensile strength and elongation at break of the films were measured by a Universal Testing Machine. The films with a dimension of 9 cm in diameter and 10N load cell. Tensile strength and% elongation at break were calculated.

3.7.1.2 WORKING PRINCIPLE

- The important function of universal testing machines is to find tensile properties.
- During this procedure, the sample object sits in the machine's jaws, and the operator attempts to elongate the sample.
- The machine demonstrates the failure point of sample and create detailed charts and graphs that show the load and stress points.
- Thus, tensile test reveals the point of greatest tensile strength, the initial point of deformation, the point of maximum deformation and the point at which the object separates into multiple pieces.

3.7.2 THICKNESS OF FILM

Simply measures 0 through 25mm with resolution to 0. 001mm.A ratchet stop or friction thimble for a constant measuring force. Measurement read out with large characters on the LCD screen.

Metric Range Order No. Accuracy
0 - 25mm 293-230 / 293-240* ±1µm
25 - 50mm 293-231 / 293-241* ±1µm
50 - 75mm 293-232 / 293-242* ±1µm
75 - 100mm 293-233 / 293-243* ±2µm

3.7.3 HARDNESS TEST USING SHORE DUROMETER

- Durometer devices determine the surface hardness of different materials,
- Durometer or hardness tester measures the depth of an indentation in the material caused by a defined force of a given geometric presser foot.
- The depth of the indentation reflects the hardness of the material.
- A general distinction is made between static and dynamic methods.
- Durometer has a reading accuracy of 0.1 hardness units.

3.8 PHYSICAL PROPERTIES

3.8.1 DETERMINATION OF WATER VAPOR TRANSMISSION RATE

- Take a neat and clean petri plate and weigh it.
- Take a known weight of calcium chloride and fill 60% off the height of the plate.
- Take the polymer material whose WVTR has to be determined.
- Cut into pieces so that it covers the petri plate and seal using a sealant
- Periodically find the weight of the petri plate until steady state is reached.
- Plot a graph of change in weight (y axis) against time (x axis).

$$WVTR = \frac{\text{Mass of water gained}}{\text{Time}\times\text{area}} = \text{flux/area } (gd^{-1}m^{-2})$$

3.9 TESTS DONE FOR APPLICATION OF FILM

3.9.1 BROWNING TEST USING SPECTROPHOTOMETER

3.9.1.1Assay Of Browning

Take 20g of frozen sample and homogenize in 40ml of distilled water for 2 minutes and centrifuged at 800rpm for 10 min in cold. One hour after homogenization, 15ml of 95% ethanol was added to 10 ml of supernatant and again centrifuged for15 min. Browning intensity was determined using spectrophotometer by taking absorbance at 440nm.

3.9.1.2 UV- VISIBLE SPECTROPHOTOMETER

UV-Visible spectroscopy is comprehended as absorption spectroscopy in the spectral region of UV-Visible spectra. Generally, light is used in visible and near-UV range. Ultraviolet and visible light are energetic enough to promote outer electrons to higher energy levels, and UV-Visible spectroscopy is usually applied to molecules in solution. The UV-Visible spectra have broad features that are of limited use for sample identification and are very useful for quantitative measurements.

The UV-Visible range approximately 400-750 nm, UV-Visible spectroscopy is useful to characterize the absorption, transmission, and reflectivity of a variety of technologically important materials, like pigments, coatings etc.

3.9.1.3 REFRIGERATED CENTRIFUGE

- Identify the speed and duration at which you wish to centrifuge samples prior to using this instrument.
- Check the rotor you intend to use and to be certain that the rotor is rated for the speed at which you would like to use it.
- If the rotor is not capable of being operated at the target speed, you will need to identify the rotor that is capable of being operated at the desired speed, and then transfer your sample to a centrifuge tube that will fit and rebalance the sample, remembering to include the lids when balancing.
- Place the rotor in the centrifuge with the two pins on the underside of the rotor forming a cross with the two pins found on the spindle of the centrifuge.
- Check the name of the rotor and confirm the target speed. Locate the correct lid for the target speed and place it beside the centrifuge.
- Place the samples into the rotor. If the samples all do not have the same mass, place samples of same mass on the opposite side of the rotor.
- Once the samples are loaded, check the rotor type again, confirm the correct lid and then screw the rotor lid onto the spindle.
- The rotor lid screws turn opposite to the normal screws. Check the proper set screw direction.
- Once the rotor is screwed, close the centrifuge lid and set the desired temperature, speed and spin time.
- When everything is set press the start button and wait for the instrument to ramp up for the desired speed.

3.9.2TEXTURE PROFILE ANALYSIS

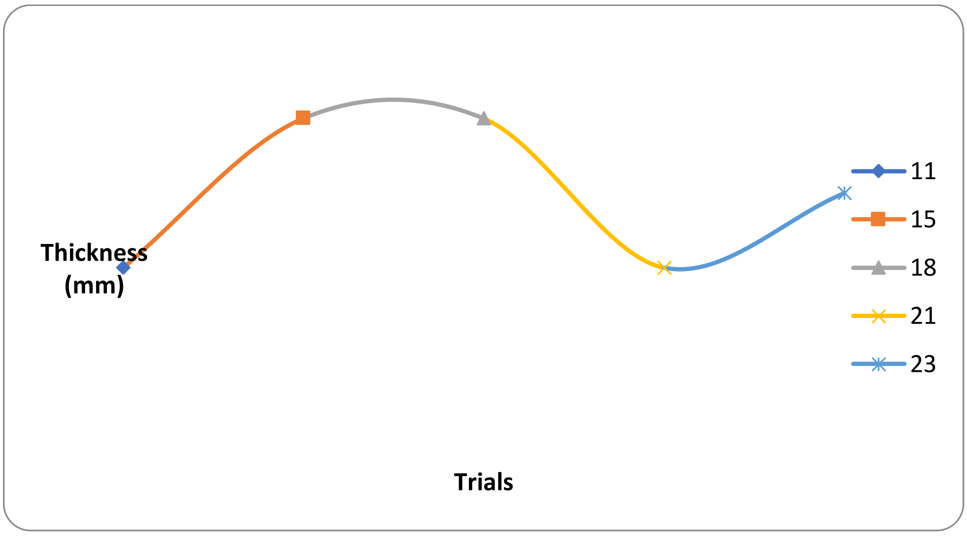
Texture was measured using a TA. HD Plus texture analyzer (Texture Technology Corp. Hamilton, Madison, USA). The probe used was TA101 ¼” rounded end probe. This device matches the perception processes of humans: masticating, sensing signals from mastication, and analyzing texture (Taniwaki, Sakurai et al. 2010). The probe corresponds to the human tooth. The speed of the probe is 22mm s^{-1} which is estimated to be the typical human mastication speed. (Roudaut, et al. 2002).

CHAPTER 3

RESUTS AND DISCUSSION

4.1 CHARACTERESTICS OF THE FILM

4.1.1 MECHANICAL PROPERTIES



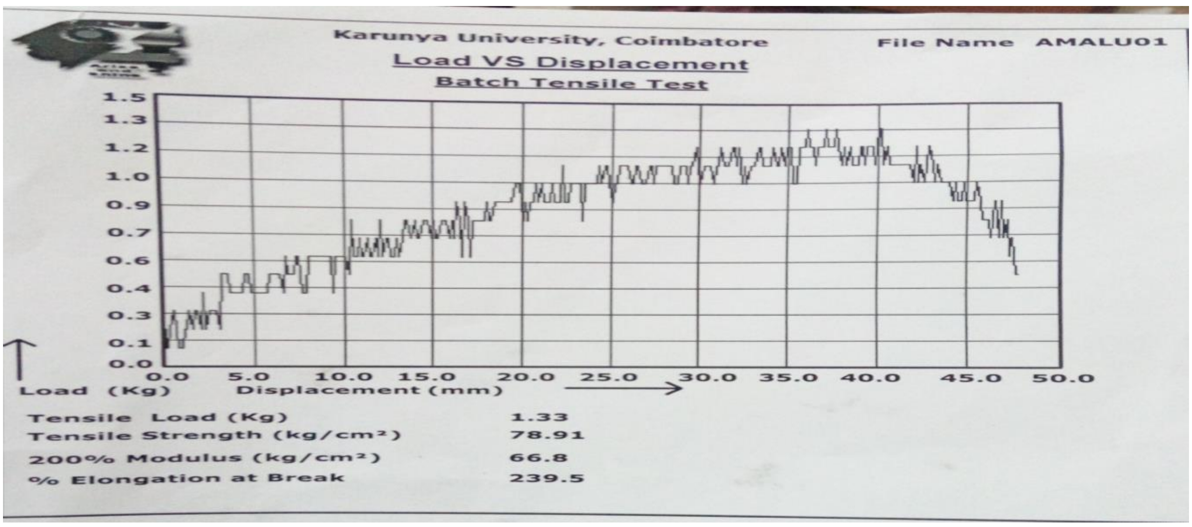
Thickness test is done using digital screw gauge. It is understood from the graph that trial 15 with ratio of casein: sorbitol: gelatin ratio as 2.5:1:2 and 18th trial with ratio of 2:1:2 have more thickness. The hardness and tensile strength are more for these films.

4.1.1.2 HARDNESS TEST

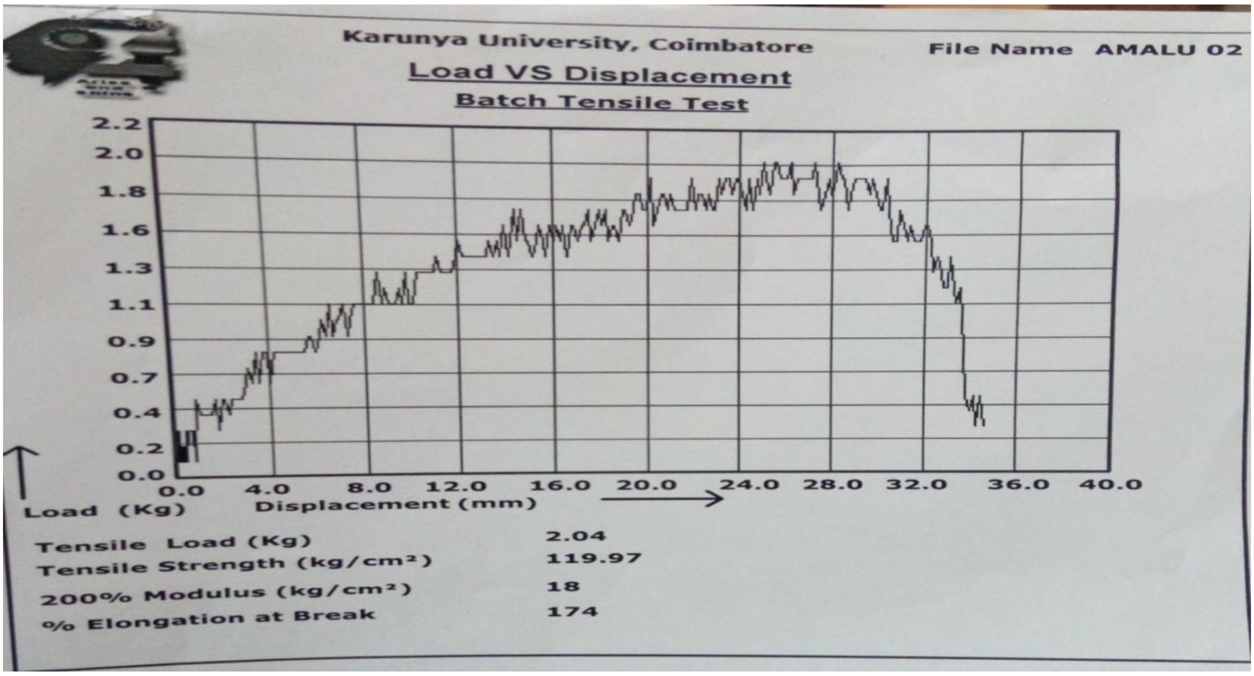
Hardness test is done using shoe durometer. The film with ratio casein: gelatin: sorbitol as 2.5:2:1 have more hardness compared to rest of the films.

4.1.1.3 TENSILE PROPERTIES

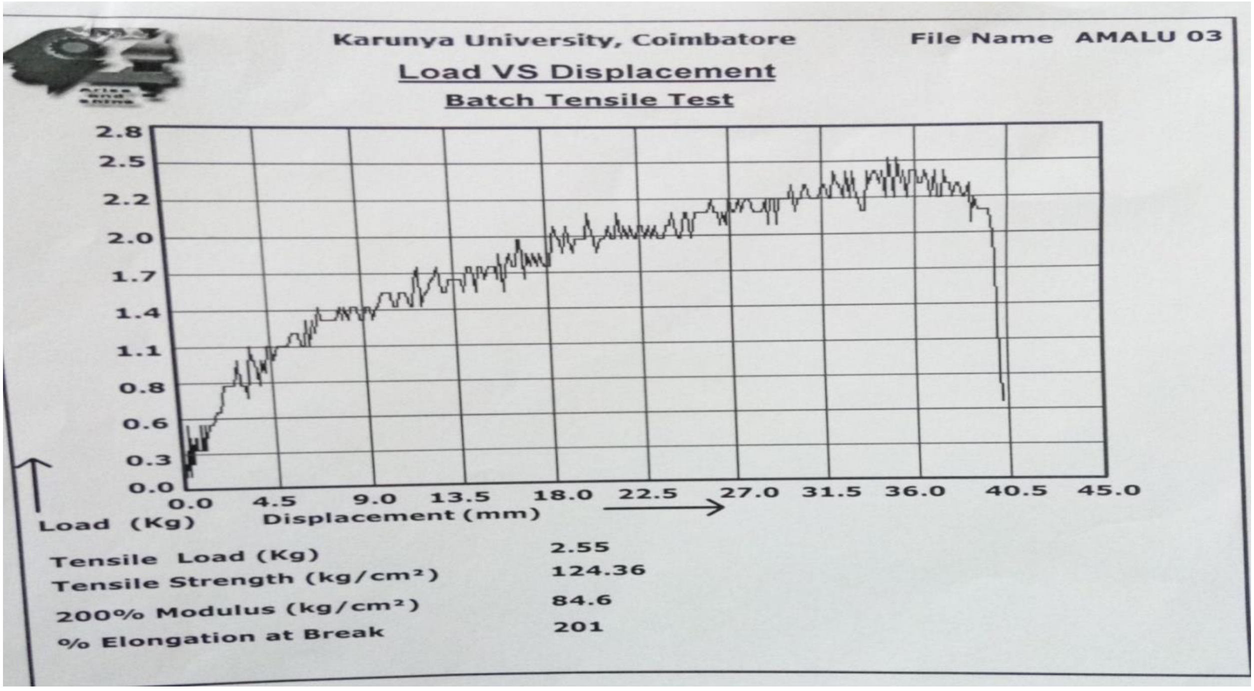
CGS5



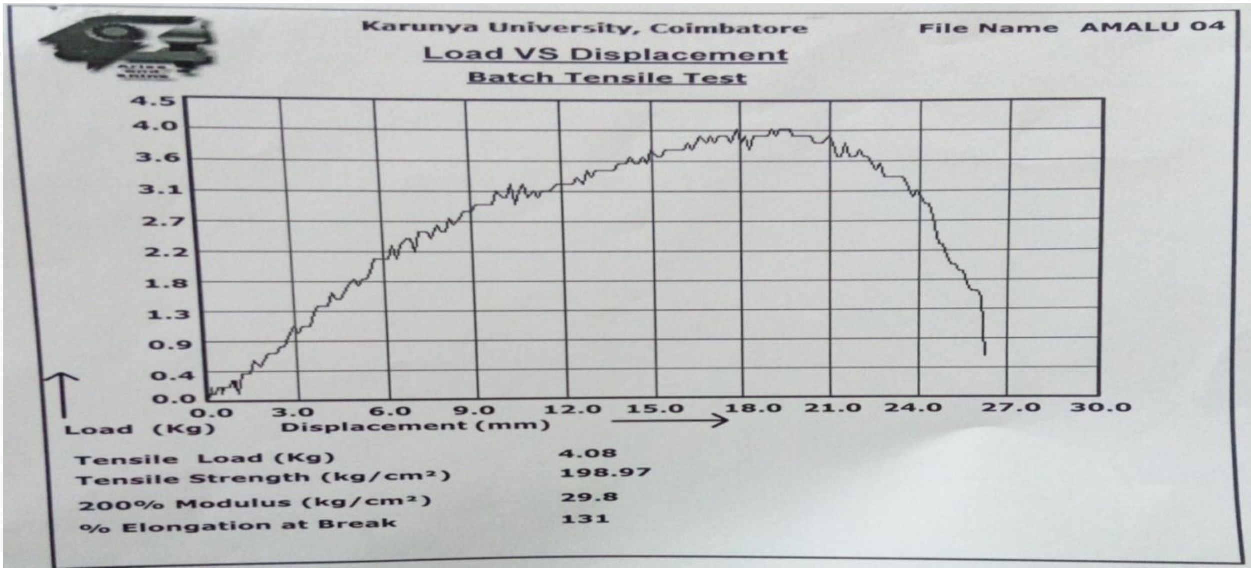
CGS4


Karunya University, Coimbatore
File Name AMALU 02
Load VS Displacement
Batch Tensile Test
Load (Kg)
Displacement (mm)
Tensile Load (Kg) 2.04
Tensile Strength (kg/cm²) 119.97
200% Modulus (kg/cm²) 18
% Elongation at Break 174

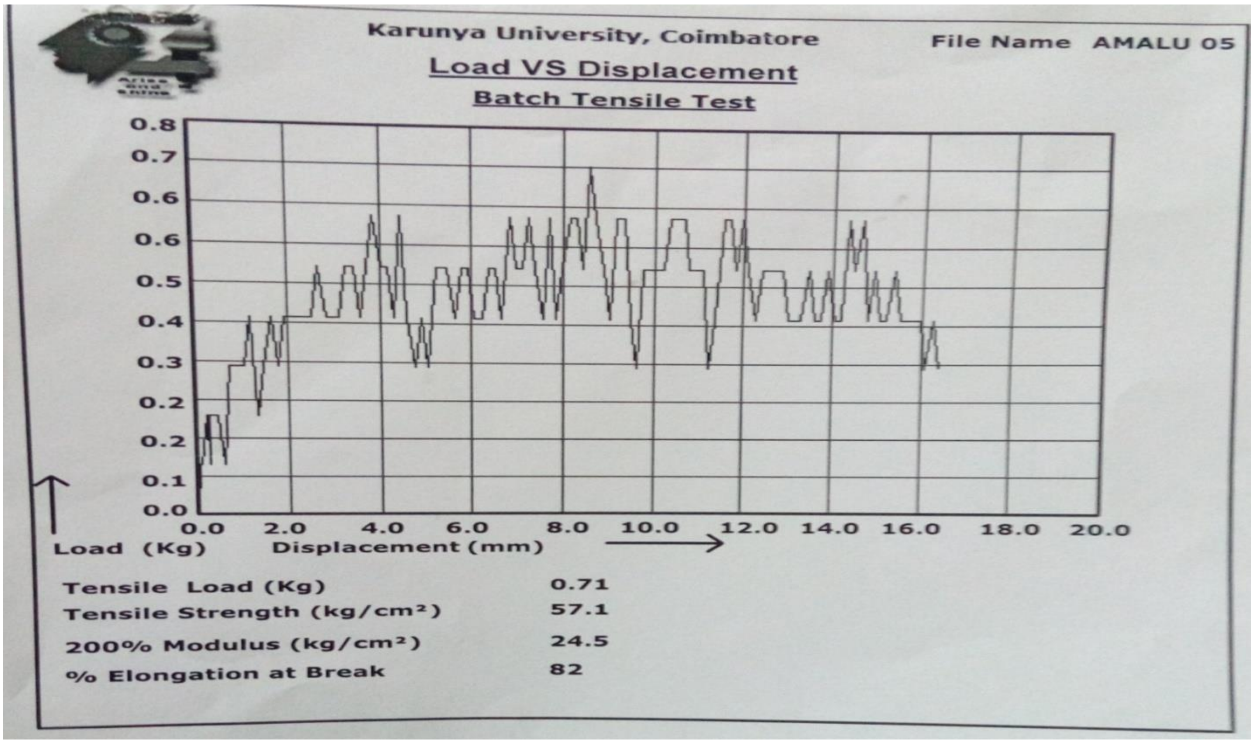
CGS3


Karunya University, Coimbatore
File Name AMALU 03
Load VS Displacement
Batch Tensile Test
Load (Kg)
Displacement (mm)
Tensile Load (Kg) 2.55
Tensile Strength (kg/cm²) 124.36
200% Modulus (kg/cm²) 84.6
% Elongation at Break 201

CGS2



CGS1



Tensile properties are measured using universal testing machine of 50KN capacity. Tensile Strength increases with increase in thickness and hardness.

4.1.2 Physical properties

CGS2 with highest hardness and tensile strength have less water vapor transmission rate

4.1. APPLICATION OF STANDARDIZED EDIBLE PACKAGE

4.1.3.1 FRESH CUT APPLES

In fresh cut apples, the browning reaction was minimized after applying the coating. Color value of apple without coating was 0.2363 and that of with coating was 0.2060.

4.1.3.2 COFFEE SACHET

The 3 in 1 coffee sachets made from casein films are dissolved completely in water and took 3 minutes for complete dissolution.

4.1.3.3 CORN FLAKES

Hardness is found to more flakes with edible coating. Hardness value of edible coated flakes is 490.465 and without coating is 327.236. Flakes with coating is less brittle and more ductile

CHAPTER 4

SUMMARY AND CONCLUSION

The outcome of this study will be focusing on the advantages of biodegradable packages over polymer packages. The packaging material made from casein is eco-friendly and recyclable. Various mechanical properties such as thickness, hardness, tensile strength, % of elongation and physical properties such as WVTR were also studied to standardize the film. Gelatin act as a thickening agent and sorbitol is used as a plasticizer. The properties of plasticizers, casein and gelatin were also studied. The standardized film which has high tensile strength and hardness have casein, sorbitol and gelatin in the ratio of 2.5:1:2. The film was formed when the amount of gelatin was increased and the amount of sorbitol was reduced. The temperature is optimized to 35^0c and the time to 20hrs after different trials. Casein edible packaging is used in three applications firstly, to reduce the browning reaction of fresh cut apples. Secondly, it is used as 3 in 1 coffee sachets which took 3 minutes to dissolve in hot water. Thirdly, it is used to reduce the sogginess of corn flakes.

CHAPTER 5

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Fig (1): Film Formed (Casein:gelatin:soritol 2.5:2:1)

Fig (2): Universal Testing Machine

Fig (3) THICKNESS TESTING MACHINE

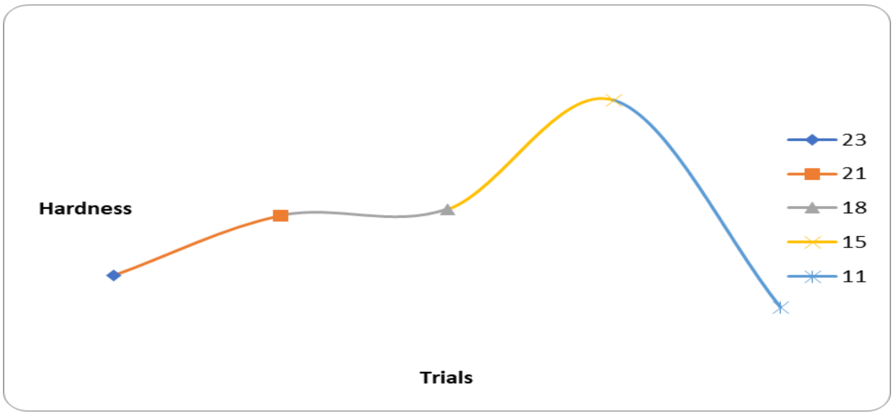


Fig (4): Graphical Representation of Hardness Test

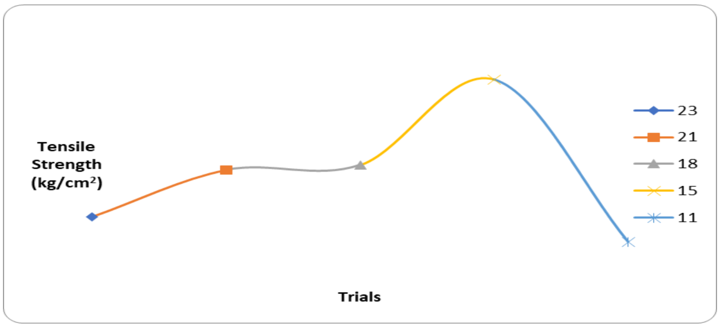


Fig (5): Graphical Representation Tensile Properties

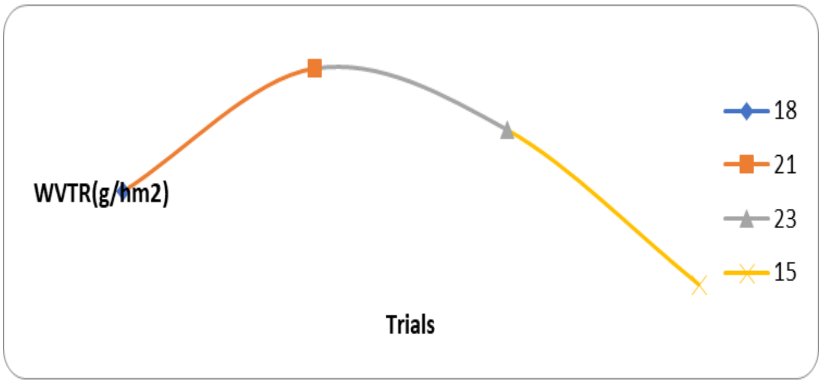


Fig (6): Graphical Representation of Water Vapor Transmission Rate

3.7.1.1 STANDARD SPECIFICATION

Machine Capacity	50Kn
Speed Range	0.001 to 1000mm/min in steps of 0.001mm/min
Crosshead Travel (excluding grips)	980mm
Throat mm	420mm

Table: 1

3.7.3.1 STANDARD SPECIFICATION

Measure range	0-100HA
Measure accuracy	≤ ± HA
Data capacity	500
voltage	3.6V
Work temperature	0^0c – 50^0c
Work humidity	20%-85%
Outline size	153mm*50mm*29mm
weight	About 169g
Sturt Diameter of the needle	1.27±0.12mm
Top plane diameter of the needle	0.79±0.03mm
Top cone angle of the needle	$(35\pm0.25)^0$
Calibration temperature	20^0c
Humidity	60%

Table: 2

4.1.1.1THICKNESS TEST

Sl.No	Trials	Thickness(mm)			Average
1	CGS1	0.29	0.28	0.19	0.25
2	CGS2	0.36	0.43	0.42	0.41
3	CGS3	0.34	0.42	0.46	0.41
4	CGS4	0.16	0.30	0.29	0.25
5	CGS5	0.38	0.38	0.25	0.33

Table: 3

Sl.No	Trials	Hardness(mm)			Average
1	CGS1	53.7	69.2	41.7	54.9
2	CGS2	79.1	71.9	71.1	74.03
3	CGS3	68.9	66.9	75.8	70.53
4	CGS4	71	66.2	66.3	67.83
5	CGS5	61.3	64	65.7	63.67

Table: 4

4.1.2.1 WATER VAPOUR TRANSMISSION RATE

SL. NO	TRIALS	WVTR(g/hm^2)
1	CGS2	
2	CGS3	
3	CGS4	
4	CGS5	

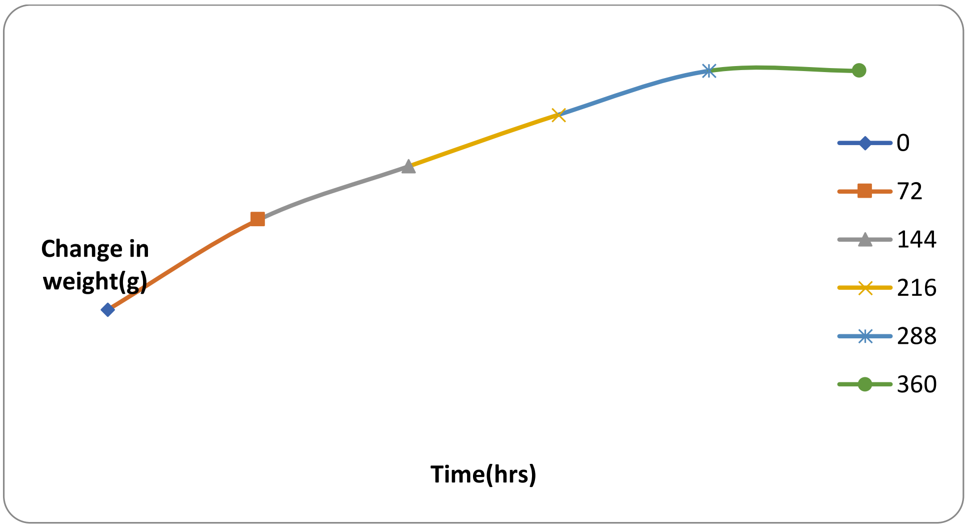
Table: 5

CHAPTER 6
APPENDIX

DETERMINATION OF WATER VAPOUR TRANSMISSION RATE

CGS2

Change in weight(g)	Time(hrs.)
41.63	0
42.6	72
42.98	144
43.77	216
44.34	288
44.34	360

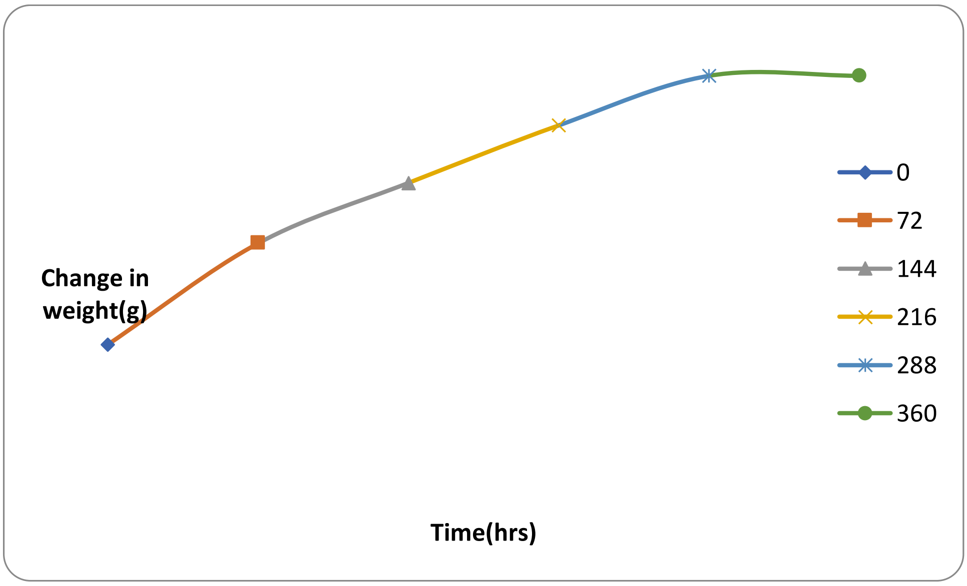


Graphical Representation of WVTR Of CGS1

$$WVTR = \frac{(44.34-41.63) \times 10^4}{72 \times 3.14 \times 20.25} = 5\ g/hm^2$$

CGS3

Change In Weight (g)	Time (hrs)
37.96	0
39.78	72
40.55	144
41.65	216
42.34	288
42.34	360



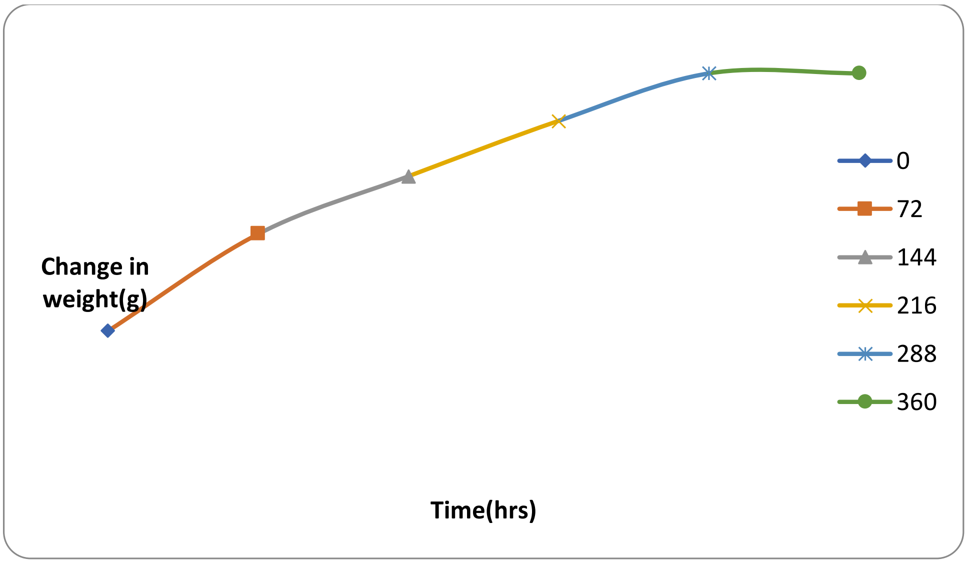
Graphical Representation of WVTR Of CGS3

$$WVTR = \frac{(42.34-37.96) \times 10^4}{72 \times 3.14 \times 20.25} = 9 \text{ g/hm}$$

CGS5

Change In Weight(g)	Time Taken (hrs.)
57.62	0
61.64	72
62.2	144
64.27	216
64.86	288
64.86	360

–

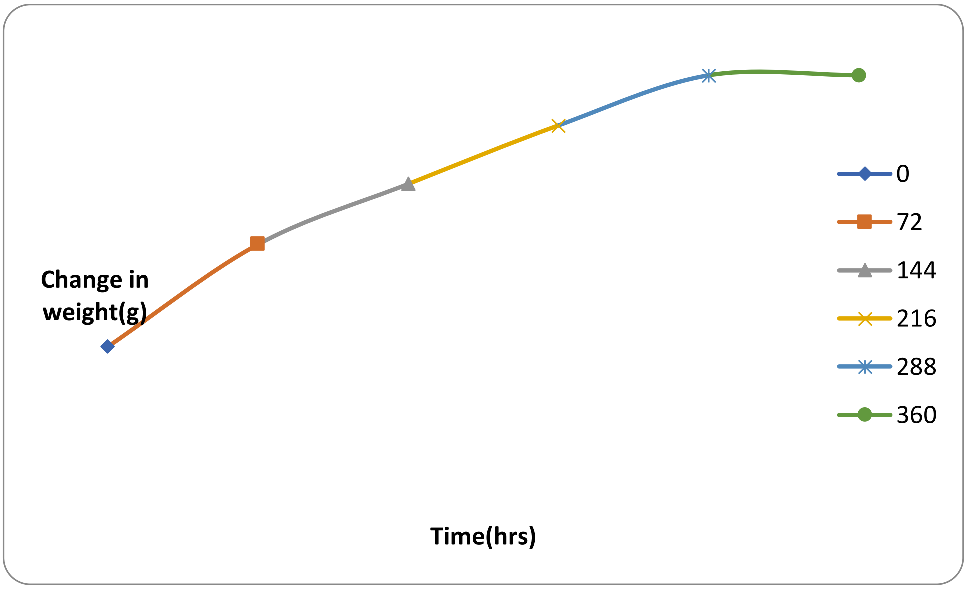


Graphical Representation of WVTR Of CGS5

$$WVTR = \frac{(64.86-57.62) \times 10^4}{72 \times 3.14 \times 42.25} = 7 \text{g/hm}^2$$

CGS1

Change in Weight (g)	Time(hrs.)
40	0
40.49	72
40.78	144
41.06	216
41.3	288
41.3	360



Graphical Representation of WVTR Of CGS1

$$WVTR = \frac{(41.3-40)\times 10^4}{72\times 3.14\times 20.25} = 2\ g/hm^2$$