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Testing and evaluation of potato starch based bio plastic

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Abstract
Plastics are hazardous to environment as they are as they are not decomposable because they are derivatives of strongly fused long sequence petrochemical grounded materials. However bio plastics can be seen as the solution to the problem. Starch is a natural biopolymer having largely two kinds of polymer glucose. In this research work bio plastic made using potato starch is tested for its various properties. However, reduced physico-mechanical properties like low tensile strength, tear strength, high rigidity, elongation at break and low moisture stability are detected in maximum of the starch-based materials. Development of starch-based bio plastic properties is being endeavoured by starch alteration. The physical and mechanical properties of the decomposable plastic have been upgraded through certain well-made procedures. Raw materials and chemicals used for production of potato starch based bio plastic are Potato, distilled water, PVA, Acetic Acid, Lecithin and glycerine. Different composition of the above ingredients were used to make bio degradable bio plastic. The bio plastic formed is tested for various parameters like thickness, hardness, gloss, water absorption test, GSM, water solubility, moisture content and biodegradability test.

Keywords: Plastic, bio plastic, starch based, properties, strength

Introduction
A plastic is a artificial or semi-synthetic substance prepared from a variety of plant polymers, such as polyethylene, that can be shaped to form when still elastic and then hardened to a solid or mildly elastic state. Bio plastics are plastics that are manufactured from natural materials (plants such as potatoes, corn, sugar, rice, tapioca, and algae) and are entirely or partly bio based, as well as biodegradable or compostable. Using microorganisms, bio plastic can be manufactured from plant by-products as well as recycled plastics (such as water bottles and other containers). Sugar derivatives such as cellulose, sugar, and lactic acid are widely used to produce bio plastics. Plastics made from petroleum or natural gas are known as fossil-fuel plastics. Bio plastics accounted for around 0.2% of the overall polymer industry (300 million tons) in 2014. Despite the fact that bio plastics are not economically feasible, research on the subject continues.

Review of Literatures
Starch can be kept in the diverse tissues of plants such as in the root of cassava plant, the stalk pith of sago, the rhizome of the potato, and seeds of corn, rice, and wheat. This starch can be collected by crushing or grinding the tubers or seeds and then mixing it up with the water in order to collect the starch as sediment. This organic starch can then be used in order to make biodegradable plastics by going through various complicated processes. Jerez *et al.* (2007) [3] Biodegradable plastics are produced from biopolymers extracted directly from biomass, with or without modification, such as starch, cellulose, and proteins. Mbey *et al.* (2012) [4] Among these, starch has presented the potential for the production of biodegradable materials because is a low-cost raw material, easily available and renewable. Fakhoury *et al.* (2012) [2] There has been increased interest in the development of biodegradable films because the environmental damage from conventional plastic packaging is increasingly evident. Ren, X. *et al.* (2003) [6] Most starch-based plastics show very poor physical properties such as tensile strength, stiffness, and elongation at the break, and poor moisture stability, which are not suitable for being used as a replacement for plastic. Bergel, B., Dias Osorio, S., da Luz, L., & Santana, R. *et al.* (2018) [1].

Said that to prevent the starch from getting affected by fungus or bacteria easily as it is an organic product; acetic acid is also used to prepare the biodegradable plastic.

Materials and Methods

The following material and method was used to formulate potato based bio plastic

1. Measure out 4.5 grams of potato starch in a beaker.
2. Add 1.3 grams of citric acid and 2 grams of Carboxymethyl Cellulose to the beaker.
3. Incorporate approximately 3.3 grams of glycerol and 1 g of PVA and mix everything together.
4. Add 70 ml of distilled water and stir at 200 rpm.
5. Place the beaker on the hot plate at 90 degrees Celsius and set it to 500 rpm for 30-40 minutes.
6. Once the solution reaches the desired consistency (honey-like consistency), turn off the hot plate.
7. Grease a Teflon sheet with a small amount of oil. This will make it easier to remove the plastic once it's dry.
8. Scoop the mixture onto the sheet and spread it out.
9. Insert the sheet into the oven at 65 degrees Celsius for 12 hours.
10. Carefully scrape the mixture off the Teflon sheet.

The bio plastic formulated above was tested for various parameters as mentioned below

Thickness Measurement (Standard Method - ASTM F2251-13)

The bio plastics thickness was measured by using micrometre. By holding the work piece between stylus and anvil, reading was directly measured.

Fig 1: Measurement of thickness using micrometre

Test for Moisture Content (Standard Method – ISO 15512)

The moisture content was estimated by measuring the weight loss of films. The bio plastics samples were cut into square pieces of 2.0 cm^2 and samples were weighed accurately. The dry film mass was documented upon drying in an oven at 110 °C till a static dry weight was attained. The moisture content was measured with five repetitions, of each film treatment:

$$\text{Moisture Content (\%)} = \frac{(W_i - W_f)}{W_i} \times 100,$$

Where,

W_i is the weight at the beginning and W_f is the final weight

Fig 2: Moisture content measurement

Water Solubility Test

Cutting into square sections of 2.0 cm^2 of the film samples, and mass was weighed accurately and recorded. The samples remained dip in 100 ml distilled water and static agitation at 180 rpm was carried out for 6 h at 25 °C. The lasting portions of the film were filtered after 6 h. samples were dried in a hot air oven at 110 °C until a final fixed weight was found. The percentage of total soluble matter (% solubility) was calculated as:

$$W_s\ (\%) = \frac{(W_0 - W_f)}{W_0} \times 100,$$

Where,

W_s is solubility in water; W_0 is the weight at the beginning of the bioplastics; and W_f is the final weight of the bioplastics.

Fig 3: Measurement of solubility

Gloss meter test (Standard Method – ASTM D2457, D523)

An instrument which is used to measure the specular reflection (gloss) of a surface is called a gloss meter. It is an tool with an incandescent light source and a photosensitive receptor to reacts to visible light. However, it has reduce appearance quality such as haze as the instrument is not sensitive to other common effects.

Many global technical principles are accessible that describe the usage and specifications for different types of gloss meter used on different types of materials.

Most common standard methods to measure the plastic materials gloss are ASTM D523 and ASTM D2457.

Fig 4: Gloss test

Standard Test Method for Specular Gloss

This test method compare with visual observations of superficial glossiness made at the corresponding angles. A black glass standard is used as a reference for comparing the specular reflectance of specimen measured gloss ratings by this test. The measured gloss ratings change as the surface refractive index changes since specular reflectance depends also on the surface refractive index of the specimen.

Biodegradability Test -Soil Burial Test (Standard Method – ASTM D5338)

For Biodegradability test specimen is cut into pieces of 4.0 cm^2. Here soil that is near the roots of plants considered rich in nitrogenous bacteria is used, 500 g of soil that is bit moist was collected and stored in a flask. Bioplastic sample was submerged inside the soil at a deepness of 2 cm and other buried at a depth of 3 cm for 15 days in atmospheric conditions. The weight of the bioplastic was measured before and after the testing. The biodegradability test was measured by Equation:

$$\text{Weight Loss (\%)} = \frac{(W_0 - W)}{W_0} \times 100,$$

Where W_o and W are the weights of samples before and after the test.

Fig 5: Soil Burial Test

Water absorption test (ASTM D570)

For the calculation of the relative water absorption rate by plastics during immersion in specified conditions Water Absorption test by ASTM D570 is used. Reasons disturbing water absorption include the plastic form, temperature, additives used, and exposure length. In ASTM D570 test method bioplastics are dried for a specified time at stated temperature and kept in a desiccator to cool for the ASTM D570 test. The samples are measured instantly after cooling. Then the material is placed inside water at 23 °C for 24 hours or until it reaches an equilibrium. Finally, the samples are removed from the water, patted dry with a lint-free cloth, and weighed.

Fig 6: Water Absorption Test

Water absorption is expressed as an increase in weight percent.

$$\text{Percent Water Absorption} = \frac{(\text{Wet Weight} - \text{Dry Weight})}{\text{Dry Weight}} \times 100$$

GSM (Standard Method – ASTM D3776)

The samples are cut by choosing the appropriate stencil seeing the type of the sample. For heavy paper (weighing above 100 GSM) stencil of the size 10 cm x 10 cm is taken and hung on one of the arms of the device. Reading is taken straight on the scale "A". If the paper or paper board is lighter in weight and reading remains below 100 GSM then stencil of size 10 cm x 20 cm is used to get more precise results and reading is taken on scale "B". At least 5 readings are taken and results are expressed in range as g/m^2.

Suitable size of paper is cut with a stencil and then weighed on a weighing balance. The weight is recorded and converted in to g/m^2 and expressed as grammage or GSM.

Fig 7: Grammage Test

Hardness test (Standard Method – ASTM D785)

The resistance of a material to deformation predominantly permanent distortion, indentation, or scratching. Hardness is

purely a comparative word and should not be confused with wear and abrasion resistance of plastic materials. For example, Polystyrene, has a high Rockwell hardness value but a poor abrasion resistance. Hardness tests can distinguish the relative hardness of dissimilar grades of a specific plastic. However, it is not useable for relating the hardness of different plastics built totally on one type of test because elastic recovery along with hardness is involved. (Shah 2007).
Two of the most commonly used tests for plastics are the Rockwell and the Durometer hardness tests. The Rockwell test is used for relatively hard plastics such as acetals, nylons, acrylics, and polystyrene. For softer materials such as flexible PVC, thermoplastic rubbers, and polyethylene, a Durometer hardness test is utilized.
The Rockwell hardness test (ASTM D785, Standard Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials) measures the net increase in depth impression as the load on an indenter is increased from a fixed minor load to a major load and then returned to a minor load. The hardness numbers derived are just numbers without units. Rockwell hardness numbers are always quoted with a scale symbol representing the indenter size, load, and dial scale used. The hardness scales in order of increasing hardness are the R, L, M, E, and K scales. The higher the number in each scale, the harder the material.

Fig 8: Apparatus for Hardness test

Results and Discussion

Bio plastic thickness

The measurement of thickness of the bioplastics at five different places is done using a micrometer, and the average is calculated.

Table 1: Values of film thickness

Replications	Film thickness (mm)
S1	0.208
S2	0.223
S3	0.215
S4	0.118
S5	0.141
Avg	0.181

The average of measured thickness of the bio plastic is found to be 0.181 mm (181 µ). The thickness of plastic bags should not be less than 50 microns, as per the regulations of the Government of India. The results demonstrate that the prepared bio plastic has a thickness of 181 microns and, which is higher as per standards and hence, can be used for making carry bags. However, several works have been reported on the thickness of bio plastics films.

Moisture Content

Moisture content is a significant factor for food processing and is anticipated to remain below the level specified level safe for storage. Five different replicas are tested by this formula and an average is calculated.

Table 2: Value of moisture content

Replications	Initial Weight W_i (in g)	Final Weight W_f (in g)	Moisture Content in Percentage (%)
S1	0.230	0.161	21.99
S2	0.408	0.310	24.019
S3	0.337	0.263	22.955
S4	0.415	0.312	22.819
S5	0.220	0.185	18.91
Avg			21.99

The moisture content for the various samples is calculated by using a formula:

$$\text{Moisture Content in (\%)} = \frac{(W_i - W_f)}{W_i} \times 100,$$

Where,
W_i is the weight at the beginning and W_f is the final weight

$$\text{For S1, Moisture Content} = \frac{(0.230 - 0.161)}{0.230} \times 100 = 21.99\%$$

Similarly, the moisture content is calculated for the other replications and then an average of all five replications are found out. An average Solubility is found to be 21.99%.
This suggests that the material retains a substantial portion of water even in its apparently dry state. This is likely due to the porous nature of the bioplastic, which can allow water molecules to be absorbed and retained within its structure.

Water Solubility

The main property that is able to differentiate the different type of starch though the film formation methods, created on starch being associated with the amylose content in the food is water solubility.

Table 3: Values of water Solubility

Replications	Initial weight W_o (in grams)	Final weight W_f (in grams)	Water solubility in (%)
S1	0.510	0.268	47.62
S2	0.359	0.179	50.08
S3	0.363	0.207	42.61
S4	0.295	0.168	43.55
S5	0.498	0.245	50.53
Avg			46.878

Solubility % is calculated by using a formula:

$$W_S (\%) = \frac{(W_0 - W_f)}{W_0} \times 100,$$

Where
W_S is solubility in water; W_0 is the weight at the beginning of the bioplastics; and W_f is the final weight of the bioplastics.

For S1, Solubility (%) = $\frac{(0.510 - 0.268)}{0.510} \times 100$

= 47.62%

Similarly, the Solubility is calculated for the other replications and then an average of all five replications are found out.
Average Solubility is found to be 46.878%
This suggests that the bioplastic's structure allows water molecules to penetrate and interact with the polymer matrix, causing it to gradually break down and dissolve. This aligns with the inherent characteristics of biodegradable polymers, which tend to exhibit water-soluble properties due to the presence of natural components that can undergo hydrolysis.

Gloss meter
Gloss is a complex attribute of a surface which cannot be completely measured by any single number. Specular gloss is used primarily as a measure of the shiny appearance of films and surfaces. Specific evaluations of gloss values are expressive only when they mention to the similar measurement procedure and identical general type of material. Table 4 shows the reading of gloss unit.

Table 4: Different values of gloss

Replications	GU
S1	16.62
S2	7.54
S3	7.6
S4	10.38
S5	10.82
Avg	10.592

The average Gloss measurement is found to be 10.592 GU. This indicates plastic has Medium Gloss.

Biodegradability Test
Microorganisms such as bacteria and fungi present in the soil can absorb and metabolize the bioplastic as a source of energy and all that remains are CO_2, water, salts and biomass.

Weight Loss (%) = $\frac{(W_0 - W)}{W_0} \times 100$,

Where,
W_o is the initial weight of sample before burial and w is the final weight after burial

Day wise weight of the bioplastic sample is recorded and at the end of sixth day it is found out that weight of plastic remains was 0.112 g
By using the data recorded in the table 4.5 and by using the formula above, the biodegradability is being calculated.

Table 5: Degradability in days

Days	Weight (g)
Day1	1.151
Day2	0.972
Day3	0.823
Day4	0.648
Day5	0.453
Day6	0.112

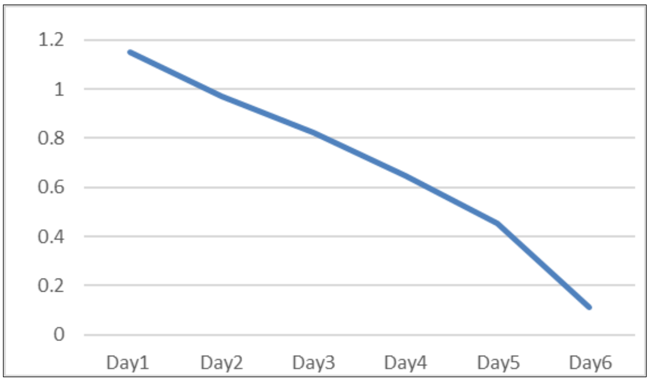

Fig 9: Graph showing biodegradability

Fig 10: Daily record of bioplastic sample

By using the formula,

Weight Loss (%) = $\frac{(W_0 - W)}{W_0} \times 100$,

= $\frac{(1.151 - 0.112)}{0.151} \times 100$

= 90.26%

Biodegradability of 90.26% was achieved in 6 days for the sample placed in the soil at a depth of 3 cm.

Water Absorption
Water absorption is used to determine the amount of water absorbed under specified conditions. Factors affecting water absorption include type of plastic, additives used, temperature and length of exposure. The data sheds light on the performance of the materials in water or humid environments.

Percent Water Absorption = $\frac{(\text{Wet Weight} - \text{Dry Weight})}{\text{Dry Weight}} \times 100$

By using above formula, the biodegradability is being calculated as shown in the table.

Table 6: Values for water solubility

Sample no	Initial weight W_o (in grams)	Final weight W_f (in grams)	Water solubility in (%)
S1	0.510	0.405	20.58

Percent Water Absorption = $\frac{(0.510 - 0.405)}{0.510} \times 100 = 20.58\%$

This show that the bio plastic material is porous in nature so, it lets water molecules to infuse and interact with the polymer matrix.

GSM

The GSM testing method involved, cutting the samples to 100cm². Using an Electrical Balance the weight of the 100cm² samples were determined.

GSM = Weight of 100 cm² x 100
= 2.617 * 100
= 216.7 GSM

200 to 300 GSM is considered good for packaging of the dry food products.

Hardness test

The potato-based bioplastic material has an average Rockwell hardness value of 41 HRC at 60 kgf load.

A moderate level of hardness suggests that the material offers a certain degree of resistance to deformation and external forces, making it potentially suitable for various applications. The observed Rockwell hardness value aligns with similar studies conducted on other biodegradable polymers, such as starch-based materials. Smith *et al*. (2020) [7] reported comparable Rockwell hardness values for their starch-based bioplastics. This indicates that the potato-based bioplastic's hardness is consistent with expectations for biodegradable materials of this nature.

Table 7: Comparison of formulated bio plastic from existing petroleum-based plastics

S. No.	Properties	Bioplastics	Polyethylene
1.	GSM	216 GSM	200 GSM
2.	THICKNESS (µ)	181 MICRON	150 MICRON
3.	Water absorbtion	20.75%	<0.01%
4.	Water solubility	46.678% soluble	Not soluble (Soluble in benzene and toluene)
5.	Gloss measurement	10.592 GU	35-97 GU
6.	Hardness	41 HRC	50 to 70 HRC
7.	Biodegradability	6 to 7 days	500 to 1000 years

We compared the formulated bioplastic with commercially available plastic based on two parameters: GSM (grams per square meter) and thickness. The results revealed that the bioplastic had a higher water absorption rate compared to polythene due to its porous nature. The addition of citric acid not only improved the material's shelf life but also enhanced its mechanical properties. Furthermore, the inclusion of PVA improved its mechanical properties.

Moreover, the bioplastic exhibited relatively high-water solubility, likely because we used food-based raw materials. This characteristic is advantageous in terms of degradability. The film also displayed a medium gloss level, and its hardness fell within the range of polythene. Notably, Formed bioplastic degraded in just one week, whereas polythene film takes thousands of years to break down.

Summary and Conclusion

Different methodologies were adopted, involving the alteration of various combinations of time and temperature. Also, the chemical composition was varied in different methods. Finally, the selected composition included distilled water (70 ml), starch (4.5 g), citric acid (1.3 g), glycerol (3.3 g), polyvinyl alcohol (1 g), and carboxymethyl cellulose (2 g). The time-temperature combination was set at 30 min and a temperature of 100 °C. With the assistance of standard test methods, we evaluated the results of various tests. The thickness of the final formulated bioplastic film measured 181 microns (µ) with a GSM of 216. The average moisture content was found to be 21.49%, while the average solubility was 46.878%. Gloss measurements yielded a result of 10.592 GU. Biodegradability reached 90.26% within 6 days. The water content percentage was 20.75%, and the average Rockwell hardness value was 41 HRC at a 60 kgf load.Subsequent testing and evaluation presented challenges in measuring and calculation, which were resolved through extensive research and knowledge acquisition. The Rockwell hardness test revealed an average value of 45.6 HRC for the potato-based bioplastic, indicating its potential for resistance to deformation and external forces, suitable for various applications. Assessed various parameters, including hardness, GSM, thickness, biodegradability, glossiness, and water absorption, comparing our bioplastic to commercially available polyethylene. The bioplastic exhibited a higher water absorption rate due to its porous nature and relatively high-water solubility due to its food-based raw materials. While it showed promise in applications like carry bags and dry food packaging. Future research should focus on reducing moisture absorption, enhancing water solubility, and reducing moisture content to engineer more eco-friendly plastics beneficial for diverse applications and the environment.

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