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MECHANICAL PROPERTIES OF STARCH-PROTEINBLEND BIOPLASTICS

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ABSTRACT

Scientists are working to find new, greener ways to replace or minimise unsustainable plastics, resulting in a growing global interest in alternative polymers. Agriculture, food processing, biomedical, and electrical industries are among the industries that are beginning to use bioplastics. Bioplastics made from starch and protein polymers have been found to have biodegradable properties,but the majority use non-sustainable starch sources. While bioplastics show biodegradation ability, it's equally important that they are also a sustainable replacement. This study combined many different starches (Potato, Tapioca, Sago and Swamp Taro) with fish gelatine to generate biodegradable bioplastics. These bioplastics were investigated for their colour, roughness, water-solubility, moisture content, transparency, tensile and elongation, hardness, and topography. Results displayed how each starch used to generate a bioplastic produced its unique mechanical data. The starch-protein blend bioplastics made with Sago and Swamp Taro were the most sustainable and showed the most promisefor bioplastic production.

KEYWORDS: Thermoset, Bioplastics, Starch, Mechanical, Characteristics, Protein, starch-protein blend.

1. INTRODUCTION

The global use of plastic packaging is increasing exponentially, due to the inherent attractive properties of conventional plastic's, such as lightweight, versatility, functionality, and relatively inexpensive nature (Saputri, A, 2016). unfortunately, these plastic products take an average of 100 years to break down (Encalada, *et al.*, 2018). As a consequence of these burgeoning industrial demands, there has been a concomitant growth in interest in sourcing alternative, bioeconomical, plastics. This increasing global pressure on use of traditional plastics is compelling scientists to find new, greener ways to produce usableplastics. However, there is still a shortage in the development of bioplastics as its high manufacturing costs lead to some limitations. So, although some industries, such as agriculture, food processing, biomedical and electrical industries, are already incorporating bioplastics into their production streams (Ashter, 2016), their endeavours are limited by the available bioplastic variants.

Starch is a suitable material for bioplastic production as a biodegradable polymer because of its low cost. Therefore, the development of bioplastics is a revolutionary advancement in solving environmental problems through renewable and degradable natural resources and the provision of more cost-effective bioplastics. (Maulida, *et al.*, 2016).

European bioplastics define a plastic substance as a bioplastic that is plastic that is either bio-based, biodegradable, or features both properties. 'Biobased' means that the commodity comes partly from plants, such as maise, sugarcane or cellulose. Bio-based does not mean biodegradable, inherently. The biodegradation property is not based on a material's resource base but its relation to its chemical structure. Thus, based on fossils, 100 % of biobased plastics can be non-biodegradable and 100 % biodegradable. Biodegradability is only unambiguous if the environment and time are defined(Doc.European-bioplastics, 2018).

According to the latest market data collected by European Bioplastics, bioplastics' global production capacity is expected to rise from approximately 2.11 million tonnes in 2019 to approximately 2.43 million tonnes in 2024, in collaboration with the nova-Institute research institute. According to the most recent Eurobarometer survey conducted by the European Commission (2013), about 80% of European customers prefer to purchase goods with minimal environmental impact.

Biobased plastics show substantial advantages over traditional plastics, making them appealing to environmentally aware consumers. Of course, this means that biobased plastics must be explained and marketed transparently: How much-biobased content is in the packaging? How much are CO2 emissions saved? Furthermore, was the biomass grown sustainably? These questions need to be addressed (Doc.European-bioplastics, 2018).

Mixtures of starch / biodegradable polymers seem to be the most promising way to develop nativestarch's mechanical and thermal properties. Starch is cheap and biodegradable, creating growing interest as a plastic component. However, starches' weak mechanical properties and water solubility have led to the development of proposed techniques for producing competitive commercial commodities, such as plasticisation or blends (Encalada, *et al.*, 2018). A paper by Halimatul from 2019 looked at the effect of sago starch and plasticiser concentrations on the mechanical properties of the produced starch films. The findings showed that both sago starch and plasticiser material significantly impacted starch films' mechanical properties. It was also found that the bioplastic films generated from taro starch are biodegradable with good mechanical properties that can replace synthetic plastics (Mrithula Shanmathy, *et al*., 2019). Thermoplastic starches' mechanical properties depend highly on the quality of moisture, plasticiser and amylose (Byun and Teck, 2014). The bioplastics produced using starch polymers have mechanical properties often inversely related to theirdegradability (Encalada, *et al.*, 2018).

With the aim to determine the effect of different starch sources on the mechanical properties of their corresponding starch-protein blend bioplastics, the objectives of this study were to (1) generate bioplastics using four different starch sources (Potato, Tapioca, Sago and Swamp Taro) and (2) to test the mechanical characteristics of each bioplastic and starch was to be accessed.

2. MATERIALS AND METHODS

2.1. Materials

Potato starch (M.P. Biomedicals LLC, Amsterdam, The Netherlands), Tapioca, Sago and Swamp Taro starches (provided by Dr. Jonay Jovani-Sancho) food-grade piscine gelatine, 200 bloom (Louis Francois, Croissy-Beauboufg, France) were used to generate bioplastics. Glycerol (EMPROVE®bio, Merck KGaA, Darmstadt, Germany) was used as a plasticiser.

Water (collected from a tributary of the river Hurley, station number: RS08H010200) and seawater (Court town, Wicklow) for the solubility.

2.2 Making bioplastics

The bioplastics were created using C. Neves, *et al*., (2020) method with the following modifications; the gelatine was heated up to 300 °C, the starch slurry with the glycerol was added, and the heat was reduced to 75 °C. Unlike that paper, the gelatine remained the same (fish), but the starches differed. The starches used were; Potato, Tapioca, Sago and Swamp Taro.

2.3 Colour

The Colorimeter Model: NO.: PCE-CSM 1 was used in this assay and was calibrated with a white background. The data was collected by placing the colourimeter over the bioplastic and pressing the start button; this was repeated three times. The colourimeter gave the readings by showing a- as green and a+ as red, b- like blue and b+ yellow.

2.4. Solubility

The dry film mass was precisely weighed and recorded after the film samples were cut into 2.0 cm2 squares. The samples underwent fixed agitation at 180 rpm for 6 hours at 25 °C while submerged in 100 mL of distilled water. They were then dried at 110 °C in a hot air oven until a final fixed weight was determined.

The percentage of total soluble matter (% solubility) was calculated as: $WS (\%) = [(W0 - Wf)/W0] \times 100$

Where W.S. is solubility in water; W0 is the weight at the beginning of the bioplastics, and Wf is the final weight of the bioplastics (Marichelvam, *et al*., 2019).

The method above was repeated with river water and seawater at 25 ℃ and their natural temperatures, 7 ℃ and 10 ℃, respectively. The temperatures were chosen based on data from the EPA website for river water and data collected onsite for the seawater.

2.5. Moisture

The materials were handled the same way as in (Marichelvam, et al., 2019), where they were accurately weighed and cut into 2.0 cm2 square pieces. The dry film mass was measured after drying for 24 hours at 110 °C in an oven until a constant dry weight was achieved. Each film treatment was used with five replications, and the moisture content was measured:

Moisture Content in (%) = [(Wi − Wf)/Wi] × 100 Wi is the weight at the beginning, and Wf is the final weight.

2.6.Roughness

The Zeiss Surfcom 130A Portable Model Handysurf Tester was used for roughness analysis. Bioplastics were laid flat on a smooth, even surface, where the sensor needle was placed over the plastic and adjusted to be lightly touched the bioplastic. The instrument was started, and the needle moved across the surface, with the instrument recording the roughness value. This was performed thrice per bioplastic to give an average for further analysis.

2.7.Transparency

Film transparency of the film samples was determined according to the method (Susilawati *et al*., 2019; Hani, 2014). The bioplastic films were cut into 1 cm × 3 cm to match the width and height of the cuvette. The films were placed to the side of the cuvette, and absorbance was recorded at 550 nmin triplicate.

$$T = A550 / x$$

Where T is the transparency, A550 is the absorbance, and x is the thickness

2.8.Tensile and Elongation

The mechanical properties of the bioplastics were determined using a 50 kN Universal Test Machine (Haida International) at a strain rate of 100 mm/min. Dog-bone shaped test pieces were produced from the various bioplastics, with reinforced grip sections to increase grip and counteract the thinness of the material.. Before starting the test, the test sample was positioned and fitted into the grips. Each sample was uniaxially extended under constant strain rate, until failure. The maximum recorded tensile strength was used to calculate the ultimate tensile strength, and the rupture point was used to determine elongation at break. The following formula was used to calculate the ultimate tensile strength:

$$\sigma_{uts} = {}^{F_{max}}/_{A_0}$$

Where: σ_{uts} = ultimate tensile strength, F_{max} = maximum force, and A_0 = initial cross-sectional area of the test specimen ((Mroczkowska, *et al*., 2021).

2.9.Hardness

The hardness of the bioplastics was measured using shore durometers A (rubber, plastic) and D(thermoplastic), with the bioplastics measuring at ~ 5 mm.

2.10. Topography

The topography of the bioplastics was measured using a Tosca Atomic Force Microscope (AFM). The bioplastics were laid flat on the stage inside the instrument, and using the software provided, the topography was seen.

2.11. Statistical analysis of data

One-way analysis of variance (ANOVA) was employed for statistical analysis, and Tukey-Kramer analysis was applied as necessary. $P \leq 0.05$ was the significance threshold. Excel and SPSS Statisticswere utilised as the analysis's software.

3. RESULTS AND DISCUSSION

3.1 Pieces of plastics

Bioplastic blends were generated comparing different starches and piscine gelatine (figure 1). Visual differences in the bioplastic's appearance were observed, and the physiochemical degradation properties were investigated. Apart from the starch types used, the composition amounts of all other elements remained constant; observed differences were due to the source of starch used.

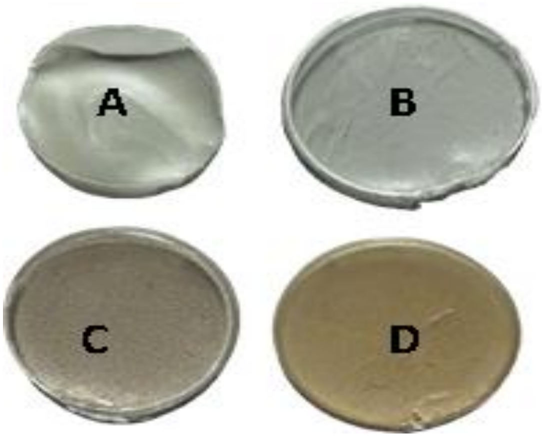

Figure 1: Displayed the bioplastics Potato (A), Tapioca (B), Sago (C), Swamp Taro (D).

3.2.Colour

The bioplastics produced were clear when looked at with no noticeable colour difference, except for the sago and swamp taro bioplastics, which were purplish and brown, respectively, figure.2. The colourimeter bioplastic results showed that the bioplastic potato and Tapioca were yellowish. The potato bioplastics produced lower value results than the literature (C. Neves, *et al*., 2020). In contrast, the Sago and Swamp Taro were yellow with red tinges. Swamp Taro's colour was similar to data froma study by (Pramodrao and Riar, 2014).

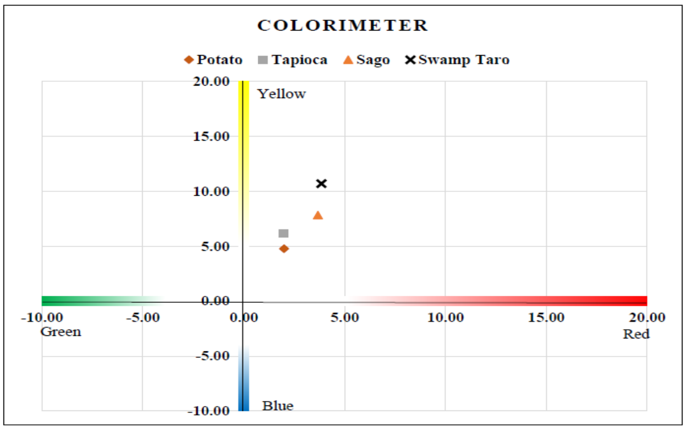

Figure 2: Colorimeter values of thermoset starch-protein blend bioplastics when different starches were used in the formulation. Values represented by dots show each starch's different colours andintensities.

The amount of fish gelatine used most likely affected the colour results as piscine gelatine has colourimeter values of 4.80 in red and 7.13 in yellow (Xavier Neves *et al*., 2019). This would have given a more yellow colour to the bioplastics. The combination of the different components most likely did affect the overall colour of the bioplastics.

3.3.Transparency

The transparency of the bioplastics was shown in the figure.3, display that the bioplastics closest to clear were produced using Tapioca starches, closely followed by Sago. Swamp Taro created the least transparent bioplastic, corresponding to its darker colour than the other bioplastics. The transparency of Swamp Taro was potentially affected by the fact that the starch wouldn't be as processed as some others. Tapioca had the lowest transparency value, 0.36, which any literature couldn't match; however, this could have been due to differences in the formulation. Adding protein in starch films also improves the transparency and the amount of amylopectin present. Amylopectin contains many long chains, which contribute to forming a compact structure that leads to a more transparent starch matrix (A. Mohammed *et al*., 2021). In addition, heat treatment, which destroys starch granules, has improved transparency (Gonzalez-Gutierrez, *et al*., 2010). Sago (1.03) and Potato (1.21), had no significant differences. Transparency may have been increased due to the addition of glycerol, which weakens hydrogen bonding and destroys the starch molecules' crystalline structure. So the degree of crystallinity decreased, and the transparency was increased. (Xin Lin Zhang, *et al.*, 2015).

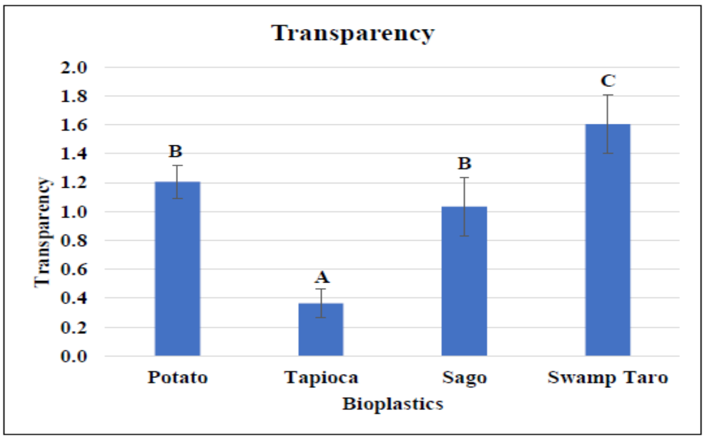

Figure 3: Transparency values of starches; Potato, Tapioca, Sago and Swamp Taro. Values represent the mean of N=4 ± standard deviation. The different subscript letter represents different significants.

The Potato had lower transparency than the other bioplastics, even though, from colour results, it had less colour than Sago. The results from the literature indicate this value was expected according to (De Azêvedo *et al.*, 2020). Finally, Swamp Taro produced the least transparent bioplastic at 1.61, giving it a different scriptor with a significant difference from all the bioplastics. These results were confirmed by other results from (Siskawardani *et al.*, 2020), which produced a value of 1.61.

3.4.Roughness

The measured surface roughness of the bioplastics yielded significant differences depending on the starch used, figure.4. Bioplastics generated with Swamp Taro and Potato starch were significantly rougher, with Ra values of 0.7640 µm ± 0.08 and 0.7467 µm ± 0.02, respectively. Compared to bioplastics developed with Sago and Tapioca, with Ra values of 0.1570 µm ± 0.01 and 0.2217 µm ±0.02, respectively.

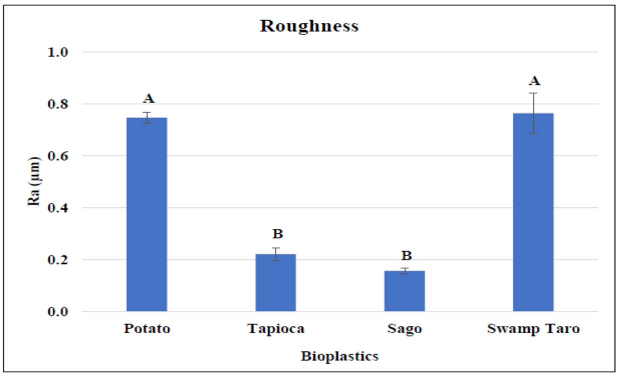

Figure 4: Roughness (µm) values of thermoset starch-protein blend bioplastics when different starches are used in their formulation. Values represent the mean of N=4 ± standard deviation. The different subscript letter represents different significants.

The potato bioplastic produced smoother bioplastics than what could be found in the literature (C. Neves, *et al*., 2020) and produced much rougher bioplastics even though the same formulation was used. The difference in the results from the literature could be due to how the bioplastics were made, as the method from C. Neves, *et al*., 2020 was slightly different to the one used here. Hydrophobic material has been reported to have higher surface roughness than hydrophilic materials (Oluwasina *etal*., 2019).

3.5.Solubility

The water solubility results were shown with the bioplastics in lab (figure.5), river (figure.6) and seawater (figure.7) at room temperature. The river and seawater were redone at their respective temperatures of 7 °C (figure.6) and 10 °C (figure.7). In these environmental water samples, they were redone in colder temperatures as the bioplastic was degraded at room temperature after 3 hours.

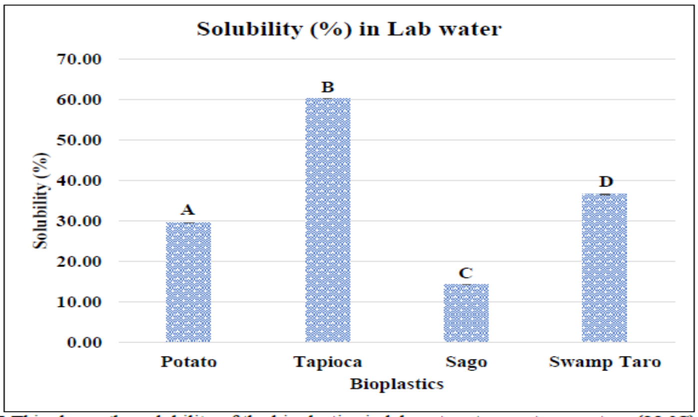

Figure 5: This shows the solubility of the bioplastics in lab water at room temperature (22 °C). Values represent the mean of N=4 ± standard deviation. Different subscript letters represent different levels of significance.

The bioplastic results showed that Sago had the lowest solubility in lab water, which was lower than the literature. The Swamp Taro was within the predicted range of 23.48 to 67.06% (Siskawardani, *et al.*, 2020). According to (C. Neves, et al., 2020), Potato was lower than expected, using the same formulation for producing bioplastics. Tapioca was found to be slightly higher than expected as the typical range was 41-66% (Baraldi de Pauli, *et al*., 2011; NGERNPIAM, 2018); in a paper by (Tongdeesoontorn *et al*., 2012), the solubility of bioplastics was found to decrease with the increase in gelatine concentration. This would explain why some of the bioplastics have lower solubility than what was found in the literature; also, some papers used a longer duration. All bioplastics were found to produce different levels of significance. The solubility of bioplastics can be an indicator of the hydrophobicity of the bioplastic. For example, since Sago had a lower water solubility, it's classed as a more hydrophobic material than Tapioca, which had the lowest hydrophobicity. This, like the moisture data, would affect the potential shelf-life of the bioplastic in terms of its water resistance.

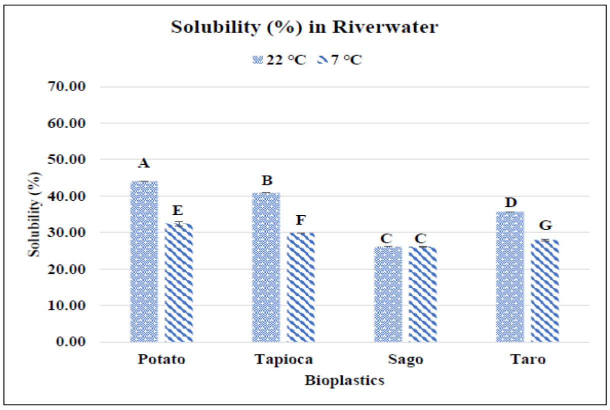

Figure 6: Shows the solubility of each starch in river water at room temperature (22 ℃) and environmental temperature (7 ℃). Values represent the mean of N=4 ± standard deviation. Differentsubscript letters represent different levels of significance.

In river water, Sago was the lowest solubility, and when the temperature was lowered to 7 °C, this remained the lowest. The solubility of Potato, Tapioca and Swamp Taro differed with temperature change, figure.6. It was also noted that when dried, unlike the samples in normal water, the river andseawater samples remained dry and brittle, whereas the lab water samples regained some of their lost weight. The solubility of the bioplastics in seawater was a more comprehensive range of 3.77% to 66.43% than in the standard lab water.

The temperature affected the solubility in seawater as the solubility at room temperature decreased when the temperature dropped to the environmental level. Tapioca and Swamp

Taro had no significant difference at room temperature; however, this changed as the temperature dropped.

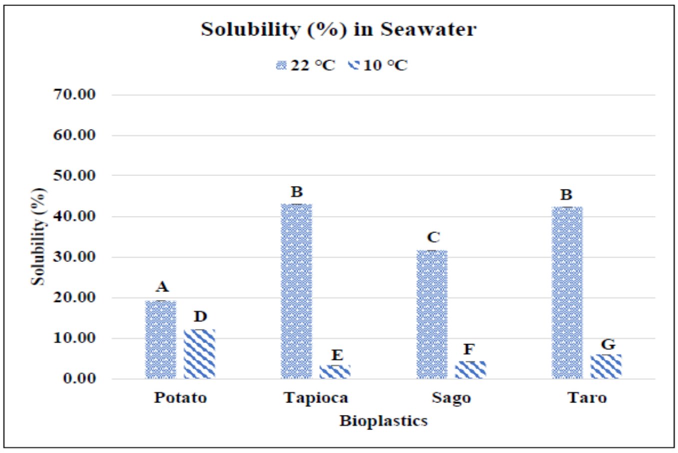

Figure 7: Shows the solubility of each starch in river water at room temperature (22 ℃) and environmental temperature (10 ℃). Values represent the mean of N=4 ± standard deviation. Differentsubscript letters represent different levels of significance.

3.6.Moisture

The figure shows some minor statistical significance between the moisture content in the starch- protein blend bioplastics, figure.8, with the moisture contents ranging from 13.87 % to 18.59 %. Sago's moisture content was within the range of 10-20 % (Halimatul *et al*., 2019), which was expected. The Tapioca and Swamp Taro were very low compared to the literature (C. Neves, *et al*.,2020; Oluwasina *et al*., 2019). The Potato bioplastic results gave the lowest moisture content. They were slightly lower than expected as they range from 15-18%, according to (Podshivalov, *et al*., 2017), which was also a starch–gelatine formulation.

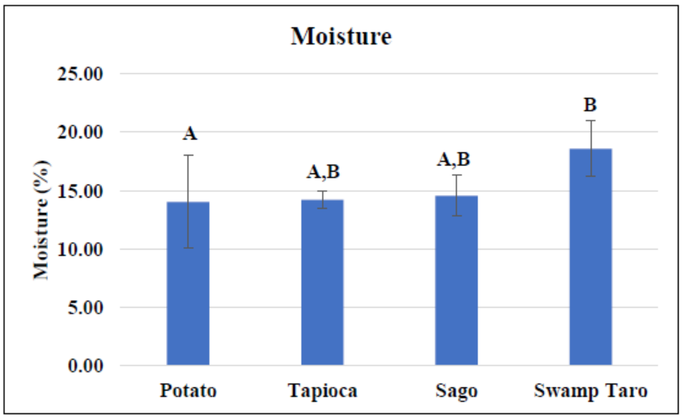

Figure 8: Moisture content (%) values of starches: Potato, Tapioca, Sago and Swamp Taro. Values represent mean N=4±standard deviation. The different subscript letters represent different significants.

The moisture content of the different starches used in the starch-protein blend formulation was completed in triplicate and ranged from 13.87-18.59%. The difference in moisture content found compared to literature could be because of the addition of fish gelatine, which has a moisture content of 16.5% (C. Neves, *et al.*, 2020), glycerol or their quantities. The moisture content of bioplastics can affect properties such as compostability, elasticity, hydrophobicity, and many other characteristics that could affect the applicability (C. Neves, *et al.*, 2020). It has been shown that moisture content also affects bioplastic shelf life (Marichelvam, *et al.*, 2019). Therefore, Potato showed minor moisture content meaning it would have a better shelf-life than Swamp Taro, which had the highest moisture content.

3.7.Tensile and Elongation

The tensile strength is the maximum load that a material can support without fracture when being stretched. Therefore, with 0.97 ± 0.13 MPa, Tapioca had the lowest ultimate tensile strength, meaning it would fracture and break faster than Sago, with 3.09 ± 0.56 MPa. The Tapioca results were similar to those from (Anugrahwidya, *et al.*, 2021), and Sago results were slightly higher than those reported by (Surya Ningrum *et al.*, 2020). Each bioplastic was allocated a unique subscript letter as none had any significance level between them, Table.1. In comparison, low-density polyethene (LDPE), used extensively for packaging and bags, has an ultimate tensile strength of 8–58 Mpa (Mroczkowska, *et al.*, 2021), making this bioplastic a bit weaker than conventional plastics.

Table 1: Ultimate tensile Strength and Elongation of the bioplastics.

Bioplastic	Elongation%	Ultimate Tensile Strength(MPa)
Potato	77.42$^{\%}$	2.52 ^{A} ± 0.47
Tapioca	165.25%	0.98 ^{B} ± 0.13
Sago	46.11%	3.1 ^{C} ± 0.56
Swamp Taro	72.01%	1.89 ^{D} ± 0.48

Ultimate tensile strength and elongation values of thermoset starch-protein blend bioplastics when different starches are used in their formulation. Values represent the mean of N=4 ± standard deviation. The different subscript letter represents different significants

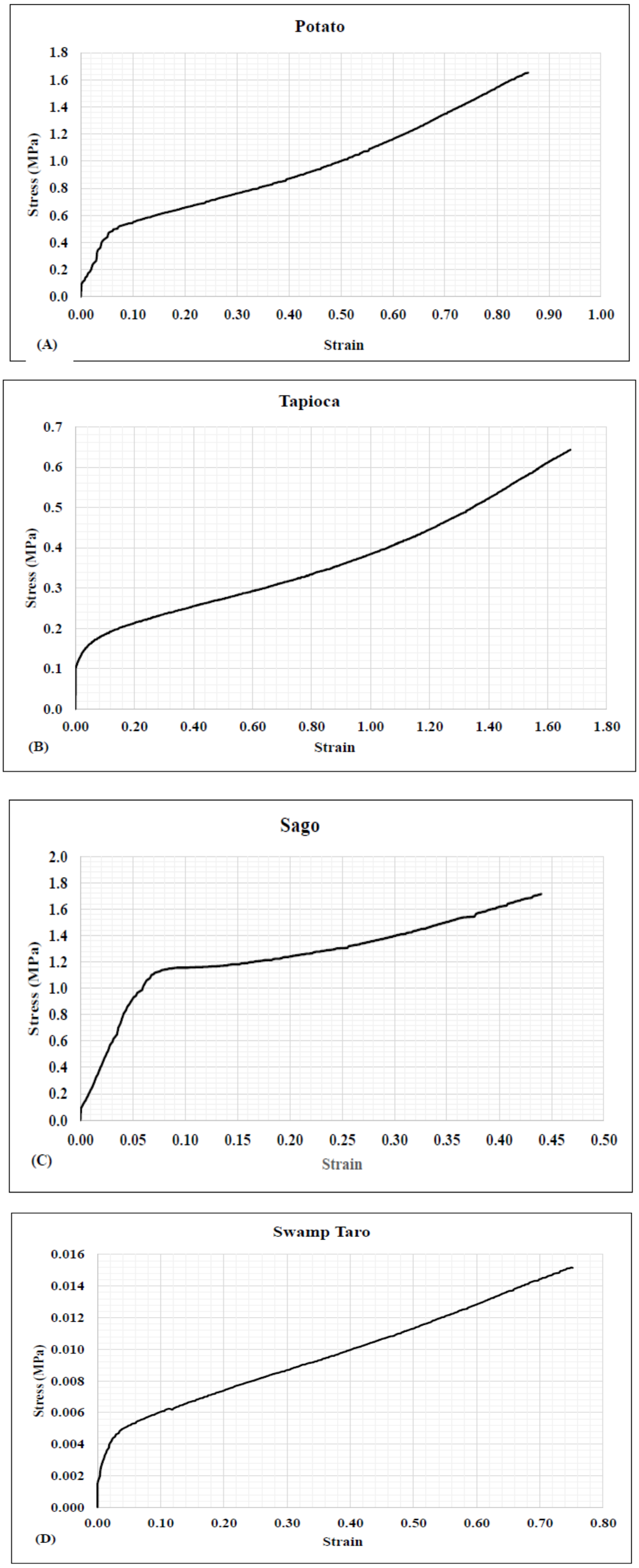

Figure 9: Stress over strain graphs of the starch-protein bioplastics (A–D).

Elongation is the length at the breaking point expressed as a percentage of its original length**.** The elongation of the bioplastics showed that Tapioca had the highest elongation (165.25%) among the bioplastics, similar to (Souza *et al.*, 2012), while Sago (46.11%) had the shortest, Table.1. Both Potato and Swamp Taro had no significant difference, with elongations of 77.42% and 72.01%, respectively. Interestingly while Tapioca had the highest elongation at the break, it had the lowest tensile strength. In contrast, Sago was the opposite, with a low elongation value and a high tensile strength value. The elongation of most bioplastics was higher than what was found in the literature; this was most likely due to the higher plasticiser content used. Higher plasticiser content has been shown to increase the viscoelasticity, giving more mobility to polymer chains. Therefore, tensile strength and elongation can be easily changed by varying the plasticiser content. Tensile strength decreases, and elongation increases with increasing plasticiser concentration (Mroczkowska, *et al*., 2021).

3.8.Hardness

The hardness of the bioplastics showed that Tapioca and Sago had no significant difference (figure.10, shore D) and were the softer of the bioplastics. While Swamp Taro was the harder of the bioplastics, this made no difference because the four starch-protein blend bioplastics were classed as amedium hardness bioplastic. When using the Shore A durometer, the values increase overall and show no significant difference between the Tapioca and Swamp Taro bioplastics but are still classed as a medium hardness plastic. Regarding literature, a study by Castro *et al*., 2021 found Potato has a shore A value of 49.43 and Tapioca can have values of 52.34-67.99 (Wahyuningtiyas, *et al*., 2018).

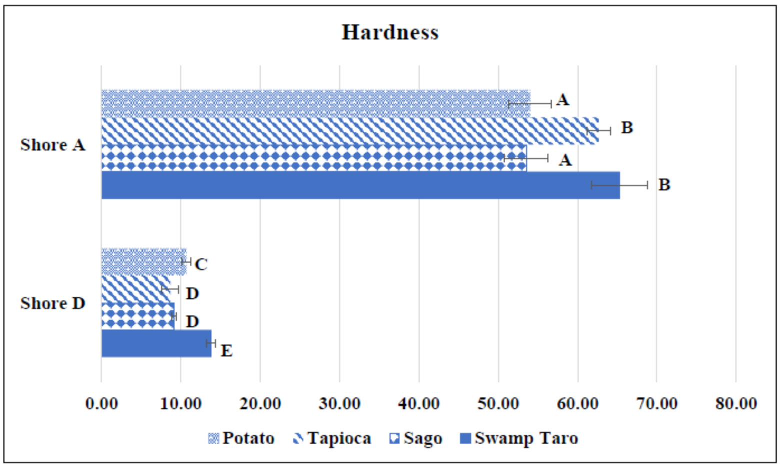

Figure 10: Hardness from Shore A and D durometers of the bioplastics. Values represent the mean of N=4 ± standard deviation. The different subscript letter represents different significants.

3.9.Topography

The topography of the bioplastics shows that while some can appear flat or smooth, the actual surface was very different, with differences in height on a microscopic level. Tapioca (figure.11.B) and Sago (figure.11.C) were the smoothest according to their roughness; however, Tapioca and Swamp Taro (figure.11.D) appear to be the smoothest. The height of the bioplastics was represented in the topography as white, red, orange, yellow, green and blue, which corresponds to a highest to lowest scale.

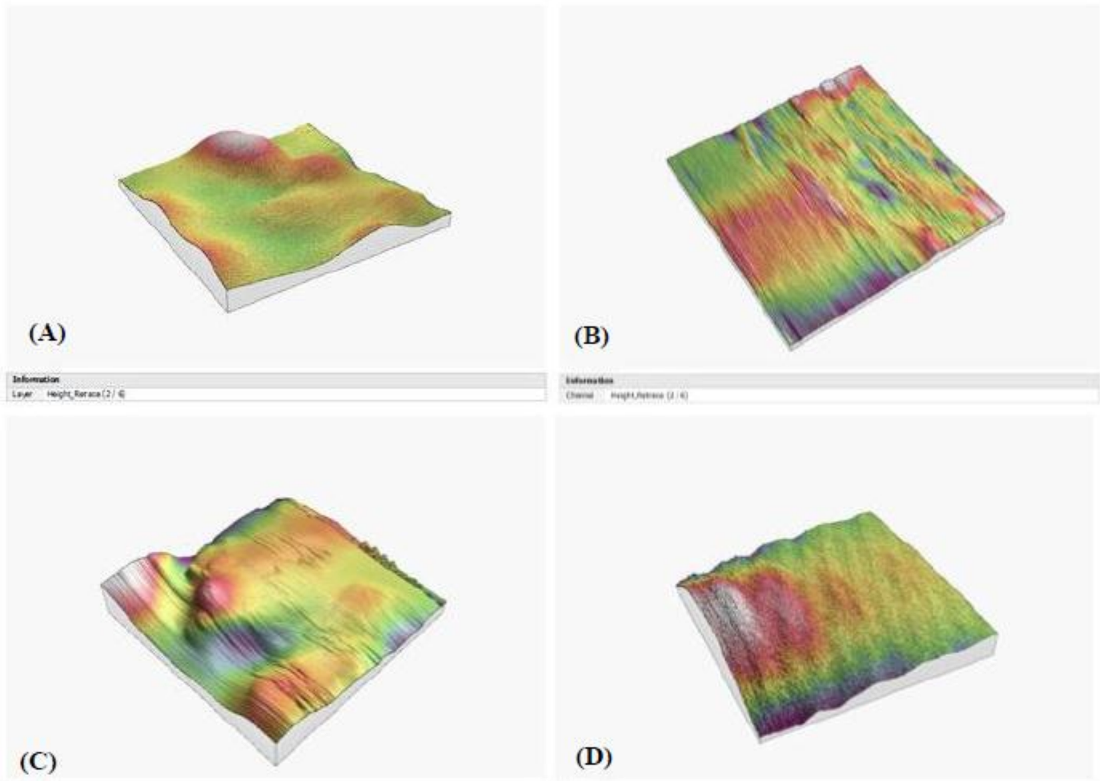

Figure 11: The topography of bioplastics (A) Potato, (B) Tapioca, (C) Sago and (D) Swamp Tarousing Tosca AFM analysis.

4. CONCLUSION

In conclusion, statistical analysis showed significant differences between the varieties of bioplastics generated over most of the assays carried out. The differences were based on which starch source was used. Depending on the end product's ideal characteristics, different starches may be more suitable.

The colourimetry of the bioplastics varied depending on the starch used. Of all the bioplastics, Sago had one of the lowest moisture contents making it have a long shelf-life, and it was the most durable against different waters for solubility. Transparency results showed that Tapioca produced the clearestbioplastics, followed closely by Sago. Swamp Taro was the least transparent, most likely due to its natural colour of the starch. Tensile strength showed that the Sago had the highest result while Tapioca had the lowest. The elongation of the bioplastics revealed that Tapioca had the highest elongation, while Potato and Swamp Taro had a good tensile strength ratio to elongation. Tapioca produced the softer bioplastic in hardness while Swamp Taro was slightly harder; all were classed as medium hardness. Finally, the topography showed how the surface of the bioplastics might not be as smooth as it originally appeared. Overall the starch-protein blend bioplastics produced show improved and, in some cases, equal mechanical properties compared to the literature.

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