

Preparation and Characterization of Thermoplastic Starch from Sweet Potato

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Abstract - Thermoplastic starch (TPS) was prepared from blends of natural sweet potato starch and polyvinyl alcohol (PVOH) at varying compositions by gelatinizing and plasticizing it with water and glycerol. The TPS samples were characterized by measuring their melting temperature, glass transition temperature, density and solubility in solvents. Their properties were found to vary with the composition of starch, PVOH and glycerol in the samples. The melting and glass transition temperatures of the TPS increased from 146 °C to 167 °C and 50.8 °C to 71.8 °C respectively, with the addition of PVOH. Addition of glycerol however reduced the melting and glass transition temperatures for both the starch-only and starch-PVOH TPS samples. The TPS samples were found to be high density plastics as their densities were greater than that of water. They were soluble in water but resistant to organic solvents. Their properties compared favourably with commercially available polymers.

Keywords - Thermoplastic, Starch, Biodegradable, Gelatinization, Bioplastics

1 INTRODUCTION

There has been a growing interest in the development of bioplastics as an alternative to the traditional petrochemical based plastics. Environmental concerns such as limiting the amount of petroleum and increasing the biodegradability of products are at the core of this growing trend towards bioplastics [1]. According to 'European Bioplastics', bioplastics are described as all plastics that are either biodegradable or bio-based, or both [2]. Among bio-based materials, starch is the most abundant; it is available in bulk in all parts of the world at low cost. Starch is renewable, readily and completely biodegradable (making it environmentally friendly and compatible). Furthermore, starch has polymeric properties and can be easily modified both physically and chemically. Consequently, starch is considered to be a very attractive raw material for the manufacture of bioplastics. Chemical and physical properties of starch have been widely investigated due to its suitability to be converted into thermoplastic materials and then to be used in different applications as a result of its known biodegradability, availability and economic feasibility [3], [4]. Thermoplastic starch (TPS) describes an amorphous or semi-crystalline material composed of gelatinized or destructured starch containing one or a mixture of plasticizers [5]. TPS can be repeatedly softened and hardened so that it can be moulded/shaped by the action of heat and shear forces. The use of starch makes it possible to replace part of a synthetic polymer with a renewable resource.

Starch ($C_6H_{10}O_5$)_n is a biopolymer of natural origin. It is composed of anhydroglucose units, linked by α -D glucosidic bonds which are arranged to form a linear chain structure (amylose) and a polymeric, branched chained structure (amylopectin) within the starch molecule. It is the end product of photosynthesis and the major storage energy in various plants in nature [6]. On industrial scale, starch is isolated from cereal grains (corn, wheat and rice) and roots (tapioca, sweet potato) [7]. Depending on botanical origin and genetic background, starch has different chemical structures and functional properties, which makes it suitable for different applications. For instance, the occurrence of different sized granules make it possible to choose the most suitable type of starch for the production of thermoplastic starch. Corn starch is the most common type used, due not only to its wide abundance but also its almost spherical shape which cause minimal disturbance in the flow during processing [8], [9]. Sweet potato starch however, presents a bright prospective in the area of thermoplastic starch production, because of its spherical shape, high amylose content of about 38%, and high starch content (6.9% - 30.7%) on wet basis [8], [10], [11]. High amylose starch produces strong films and is therefore, suitable for making thermoplastic starch materials.

An important property of starch is its semi-crystallinity. The crystalline structure of starch can be disrupted in the presence of a plasticizer, heat and shear [12], [13], [14], [15]. This results in a de-crystallized or gelatinized starch/plasticizer mixture. Therefore, in order to produce TPS products, it is necessary to disrupt the starch granules and melt the crystalline structure. This is achieved by subjecting the starch molecules to heating and shearing effects in the presence of excess water or any other plasticizer able to form hydrogen bonding with starch hydroxyl groups. This causes the starch molecules to breakdown, (resulting in the destruction of the crystalline structure) and gets gelatinized and plasticized with water. As a result, a homogenous amorphous mass (starch melt) is formed. When the gelatinized starch/water mixture is exposed to the atmosphere, the water that is present in the

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mass expands into steam due to sudden drop in pressure and the thermoplastic starch is formed. Plasticizers (such as glycerol, sorbitol, propylene glycol, ethylene glycol, polyethylene glycol, triethyl citrate and triacetate) can be used instead of water to impart flexibility and ductility to the TPS product [16]. TPS is able to flow at high temperatures and can be processed on conventional plastic fabrication equipment such as twin screw extruder where it goes through a thermo-mechanical process simultaneously in the presence of water and plasticizer thereby, causing the physical structure of the processed starch to be different from that of native form [17].

Native starch has poor mechanical properties. Also, it is hydrophilic (water soluble) and cannot be processed by wet-based routes without being degraded [18]. Therefore, it needs to be blended with high performance synthetic polymers such as polyvinyl alcohol (PVOH), polylactic acid (PLA) and polybutylene succinate (PBS) to improve the mechanical properties of the thermoplastic starch to compete with the conventional plastics. Blending or grafting of starch with synthetic polymers has been suggested by several authors as a suitable route in improving properties [13], [19], [20], [21], [22], [23], [24], [25], [26], [27], [28], [29], [30]. Thermoplastic starch have been found to have useful performance properties which finds application in areas such as: agricultural mulch film, biomedical materials, shopping bags, food waste films, coated paper, consumer packaging materials and landfill cover etc.

The main objective of this work was to investigate the feasibility of producing thermoplastic starch from sweet potato and to characterize the thermoplastic starch by determining its physical properties such as: density, solubility in solvents, and transition temperatures (melting and glass transition).

2 MATERIALS AND METHOD

2.1 Materials and Reagents

Freshly harvested sweet potato (*Ipomea batatas*) purchased from a local farm was used as the source of starch. High grade polyvinyl alcohol powder (manufactured by Harlow Chemical Co. Limited, Essex Harlow, UK) was purchased from a local chemicals vendor. All reagents used were of analytical grade and were used without further purification. They include; glycerol (wt per ml: 0.124 to 1.261g, BDH limited Poole, England), ethanol (wt per ml: 0.788 to 0.790g, BDH limited Poole, England), xylene (wt per ml: 0.855 to 0.866g, BDH limited Poole, England), distilled water, paraffin oil, and benzene. They were purchased from a local chemicals vendor.

2.2 Experimental Method

2.2.1 Extraction of Starch from Sweet Potato

Starches were isolated according to the method of Collado and Corke [31]. Freshly harvested sweet potato were washed thoroughly, immersed in cold water for one hour, peeled and sliced. The slices were grated finely, soaked in distilled water, washed extensively and sieved through a cheese cloth. The filtrate (starch-water mixture) was passed through a 250 μm

sieve and left for 2 hours for the starch molecules to settle. The supernatant was then decanted, leaving behind the starch paste (as sediment) which was dried in a laboratory oven at 50 °C for 5 hours. The dry starch lump was gently crushed into powder. The powdered starch was stored in an air tight bag to prevent moisture and contamination.

2.2.2 Formulation of TPS Samples

Four samples of TPS (labeled as A, B, C and D) were prepared. The composition of the samples include; A (100 wt% starch), B (75 wt% starch, 25 wt% PVOH), C (100 wt% starch plus glycerol) and D (75 wt% starch, 25 wt % PVOH plus glycerol). Sample A consisted of 20g of sweet potato starch mixed with 20ml of cold distilled water in a 250ml beaker. The starch slurry was stirred vigorously and the initial temperature was taken. The beaker was clamped to a retort stand, and heated while the starch slurry was stirred continuously until it got plasticized. The temperature at which the starch was plasticized was recorded. Sample B consisted of 15g of starch was mixed with 5g of PVOH, and 20 ml of cold distilled water was added to the mixture in a 250 ml beaker. The mixture was stirred until there was proper mixing. The beaker was clamped to a retort stand, and the mixture was then heated and stirred continuously until it got plasticized. The temperature at which the mixture was plasticized was recorded. Sample C consisted of 20g of starch mixed with 15ml of glycerol in a 250 ml beaker. The mixture was stirred vigorously until there was proper mixing of the two substances. The beaker was clamped to a retort stand, and the mixture was heated and stirred continuously until it got plasticized. The temperature at which the mixture was plasticized was recorded. Sample D consist of 15g of starch mixed with 5g of PVOH, and 15 ml of glycerol was added to the mixture in a 250 ml beaker. The mixture was stirred until there was proper mixing. The beaker was clamped to a retort stand, and the mixture was then heated and stirred continuously until it got plasticized. The temperature at which the mixture was plasticized was recorded. After plasticization, the four samples were placed in different dishes whose inside surfaces have been lubricated with oil and dried in a tray dryer at a temperature of 60 °C for 8 hours to remove any moisture present in the samples.

2.2.3 Characterization of the TPS Samples

The TPS samples prepared were characterized by measuring their melting temperatures, glass transition temperatures, densities and solubility in solvents.

2.2.3.1 Measurement of Density

The densities of the TPS samples were measured using the orifice can. A portion of sample A was cut and weighed using a digital weighing balance, the mass was recorded. The orifice can was filled with distilled water until the water starts flowing through the orifice. When the water stopped flowing, the weighed sample was dropped into the water in the orifice can and the water displaced was collected in a beaker whose

mass has been measured. The mass of the beaker and the water displaced was also measured using a weighing balance. The mass of water displaced (upthrust) and the volume of water displaced was determined. The density of the sample was then determined by (1).

$$\text{Density of TPS sample} = \frac{\text{mass of TPS sample in air}}{\text{volume of water displaced}} \quad (1)$$

The same procedure was repeated for all the samples.

2.2.3.2 Test for Solubility in Solvents

10ml each of cold water, hot water, benzene solution, ethanol solution and xylene solution were added to 1g of sample A in different test tubes, and allowed to stay for 24 hours. The degree of solubility of the sample in the solvents was recorded. The same procedure was repeated for samples B, C and D; and in each case, the degree of solubility in the solvents were recorded.

2.2.3.3 Determination of Melting Temperature

The melting temperatures of the TPS samples were measured by using the WRR melting point apparatus. The apparatus has a melting point measuring range of 40 – 280 °C, and accuracy of ± 0.5 °C. The WRR melting point apparatus can measure three samples at the same time, and calculate the average value of initial and final melting point automatically. A portion of sample A was crushed and filled in three thin-walled capillary melting point tubes. The capillary tubes were 120 mm long with about 1 mm inside diameter and sealed at one end. The capillary tubes with the densely packed sample were placed in the apparatus. The heating of the samples in the apparatus was achieved by a u-shaped tube as oil bath and methyl-silicone oil as heating medium. The heating and cooling of the oil bath is achieved through an electronic heating block and cooling fan. The apparatus is equipped with digital thermometer, magnifier lens, temperature control program/system and digital display of initial and final melting temperatures. The temperature control system allows the start temperature to be set and a linear heating rate to be selected from a set of four steps (0.5, 1.0, 1.5, and 3 °C/min). One capillary tube filled with sample A was first heated at a rapid rate (3 °C) to roughly identify the likely melting range of the sample. The heating was repeated with the three capillary tubes loaded with the sample and the start temperature was set at 20 °C lower than the melting point of the sample. The samples were then heated at a slow rate of 1 °C/min until the samples were observed to melt through the magnifier. The initial and final melting temperatures were observed and recorded. The average of the two temperatures is reported as the melting temperature of the sample. The same procedure was repeated for samples B, C and D.

2.2.3.4 Determination of Glass Transition Temperature

The glass transition temperature (T_g) of the samples were measured empirically by using the correlation proposed by [32] and presented by [33]. This is shown in (2).

$$\Delta H_{\text{fus}}/C_p^L = 0.55(T_m - T_g) \quad (2)$$

According to Chickos et al. [34], the heat of fusion (ΔH_{fus}) of the samples can be calculated based on atomic group contribution as presented in (3), (4), (5), and (6).

$$\Delta H_{\text{fus}} = T_m \Delta S_{\text{fus}} \quad (3)$$

$$\Delta S_{\text{fus}} = a + b \quad (4)$$

$$a = 35.19N_R + 4.289(N_{\text{CH}_2} - 3N_R) \quad (5)$$

or $a = 0$, if there is no non-aromatic ring in the molecule

$$b = \sum_{i=1}^{n_g} (N_g)_i (\Delta_s)_i + \sum_{j=1}^{n_s} (N_s)_j (C_s)_j (\Delta_s)_j + \sum_{k=1}^{n_f} (N_f)_k (C_t)_k (\Delta_s)_k \quad (6)$$

The correlation proposed by [35] was used to calculate the liquid specific heat capacity of the samples at constant pressure (C_p^L). The correlation is presented in (7).

$$C_p^L = \sum_{i=1}^n N_i \Delta C_{p_i} + 18.83m \quad (7)$$

3 RESULTS AND DISCUSSION

The plasticization temperature of the four TPS samples was obtained as follows; A (86 °C), B (90 °C), C (118 °C) and D (106 °C) respectively. Other results obtained from this study are presented in Tables 1 and 2. Table 1 shows the melting temperature, glass transition temperature and density of the thermoplastic starch samples. Table 2 shows the degree of solubility of the thermoplastic starch samples in different solvents. The literature data for physical properties of some widely used polymers is presented in Table 3.

From the data presented in Table 1, the melting temperature of samples A, B, C and D are 146 °C, 167 °C, 83.9 °C and 94.5 °C respectively. This result shows that the melting point of sample A (100 wt% starch), is lower than that of sample B (75 wt% starch, 25 wt% PVOH). This difference in the melting temperature of the two samples can be attributed to the blending of starch in sample B with PVOH which has a high melting point of 230 °C. The presence of the polar side group (OH) in the PVOH molecule leads to significant intermolecular bonding forces between starch molecule and PVOH molecule, resulting in a higher melting temperature [36], [37]. The melting temperature of samples C and D (83.9 °C and 94.5 °C respectively) were much lower than those of samples A and B. This trend may be attributable to the addition of glycerol as a plasticizer to the samples. Hence, sample A and B will withstand higher service temperature than samples C and D. It is also evident from Table 1, that samples A and B melt at a higher temperature than low

density polyethylene (LDPE) and high density polyethylene (HDPE) which have melting temperatures of 115 °C and 137 °C respectively (Table 3). Hence, these TPS samples will withstand higher service temperature than LDPE and HDPE.

The glass transition temperature of samples A, B, C and D are presented as 50.8 °C, 71.8 °C, -11.3 °C and -1.8 °C respectively. The implication of these results is that, at room temperature (25 °C), samples A and B will exist in the glassy (amorphous) state, since their glass transition temperatures (T_g) are far above room temperature. Therefore, these thermoplastic starch samples are expected to exhibit glass-like characteristics such as: brittleness, hardness, high flexural modulus (high stiffness), rigidity, optical transparency, rapid deformation under applied stress and less resistance to reagent attack. On the other hand, the glass transition temperature of samples C and D (-11.3 °C and -1.8 °C respectively) are lower than room temperature, hence this TPS samples will exist in the rubber-like state at room temperature. This indicates that samples C and D are crystalline in nature, hence at room temperature these samples will exhibit toughness, strength, flexibility, long range elasticity, resistance to reagent attack and high crystallinity. The low glass transition temperature of samples C and D is as a result of the addition of a plasticizer (glycerol) to these samples. The glycerol molecule eases the movement of the polymer chains by pushing them further apart, thus increasing the free volume [38]. This effectively reduces the inter-chain forces within the molecule and hence, T_g is depressed

From Table 1, the densities of samples A, B, C and D are presented as 1.705 g/cm³, 1.373 g/cm³, 1.148 g/cm³ and 1.095 g/cm³ respectively. This indicates that the TPD samples are high density plastics; this is based on the fact that these plastics have densities greater than that of water (1.0 g/cm³). These results are at variance to the results obtained by Griffin [39] for LDPE and HDPE which had densities of 0.917 g/cm³ and 0.965 g/cm³ respectively.

From Table 2, samples A and B were slightly soluble in cold water. All of the four samples were highly soluble in hot water. This result is as a result of the highly hydrophilic nature of starch and PVOH [40]. Hence, the samples will readily disintegrate when they come in contact with water. Samples A, B, C and D were resistant to the organic solvents, and hence they can be used in handling such solvents.

4 CONCLUSION

The research work showed that thermoplastic starch (TPS) can be produced from natural sweet potato starch. The TPS was prepared by blending sweet potato starch with different compositions of PVOH, and then gelatinizing it with glycerol and water. The samples of TPS produced were characterized and the results were compared with the literature data for some widely used polymers. The physical and thermal

properties of the TPS were found to vary with the composition of starch, PVOH and glycerol in the sample. The melting and glass transition temperatures of the TPS increased with the addition of PVOH. Addition of glycerol reduced the melting and glass transition temperatures of both the starch-only and starch-PVOH TPS samples. TPS from sweet potato starch was found to be high density plastic, with density greater than that of water. The TPS samples were subservient to water attack, but resistant to organic solvents. TPS from sweet potato possess good performance properties comparable to the conventional plastics. Commercial production of sweet potato based thermoplastic starch can therefore be encouraged in geographical locations where its production is abundant and long-term storage systems are lacking.

5 ABBREVIATIONS

| | |
|---------------------|--|
| ΔH_{fus} | Latent heat of fusion of sample at T_m (cal/mol). |
| CP,L | Liquid specific heat capacity at constant pressure of sample (cal/mol K). |
| T_m | Melting temperature of the sample (K). |
| T_g | Glass transition temperature of the sample (K). |
| ΔS_{fus} | Entropy of fusion at T_m (J/mol K). |
| N_R | Number non-aromatic rings. |
| N_{CH_2} | Number of -CH ₂ -atomic groups in non-aromatic ring required to form a cyclic paraffin of the same ring size. |
| n_g | Number of different non-ring or aromatic C-H atomic groups bonded to other carbon atoms in the molecule of interest. |
| $(N_g)_i$ | Number of C-H atomic groups of type i bonded to other carbon atoms in the molecule of interest. |
| n_s | Number of different non-ring or aromatic C-H atomic groups bonded to at least one functional group or atom in the molecule of interest. |
| $(N_s)_j$ | Number of C-H atomic groups of type j bonded to at least one functional group or atom in the molecule of interest. |
| n_f | Number of different functional groups or atoms in the molecule of interest. |
| $(C_s)_j$ | Coefficient for C-H atomic group j bonded to at least one functional group or atom in the molecule of interest. |
| C_t | Coefficient for the functional group or atom k in the molecule of interest, where t is total number of functional groups or atoms in the molecule. |
| $(\Delta S)_{ij,k}$ | Contribution of the atomic group or atom i, j, k to the entropy of fusion (J/mol K). |
| n | Number of different atomic groups in the compound. |
| N_i | Number of atomic groups i in the compound. |
| ΔCP_i | Numeric value of the contribution of atomic element i (J/mol K). |
| m | Number of carbon groups requiring an additional contribution, which are those that are joined by a single bond to a carbon group, which in turn is |

connected to a third carbon group by a double or triple bond.

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TABLE 1
PHYSICAL PROPERTIES OF TPS SAMPLES

| Sample | Melting Temperature, T_m ($^{\circ}C$) | Glass Transition Temperature, T_g ($^{\circ}C$) | Density, ρ ($g\ cm^{-3}$) |
|--------|---|--|-------------------------------------|
| A | 146 | 50.8 | 1.705 |
| B | 167 | 71.8 | 1.373 |
| C | 83.9 | -11.3 | 1.148 |
| D | 94.5 | -1.8 | 1.095 |

TABLE 2
DEGREE OF SOLUBILITY OF TPS SAMPLES IN SOLVENTS

| Sample | Degree of Solubility | | | | |
|--------|----------------------|----------------|-----------|-----------|-----------|
| | Cold Water | Hot Water | Benzene | Ethanol | Xylene |
| A | Slightly Soluble | Highly Soluble | Insoluble | Insoluble | Insoluble |
| B | Slightly Soluble | Highly Soluble | Insoluble | Insoluble | Insoluble |
| C | Slightly Soluble | Highly Soluble | Insoluble | Insoluble | Insoluble |
| D | Slightly Soluble | Highly Soluble | Insoluble | Insoluble | Insoluble |

TABLE 3
PHYSICAL PROPERTIES OF SOME WIDELY USED POLYMERS

| Sample | Melting Temperature, T_m ($^{\circ}C$) | Glass Transition Temperature, T_g ($^{\circ}C$) | Density, ρ ($g\ cm^{-3}$) |
|--------|---|--|-------------------------------------|
| LDPE | 115 | -110 | 0.917 |
| HDPE | 137 | -90 | 0.965 |
| PVC | 200 | 87 | 1.400 |
| PVOH | 230 | 85 | 1.310 |

Source: Griffin [39]