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RESEARCH ARTICLE

Fabrication and Characterization of Starch-Based Biodegradable Polymer with Polyvinyl Alcohol

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Abstract

Starch is a renewable, biodegradable and low-cost natural biopolymer with high availability. Starch was extracted from *Solanum tuberosum* (potatoes) and its preliminary properties were studied. The structure of extracted starch was characterized by Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). A series of starch-based biodegradable blends, with polyvinyl alcohol (PVA) were prepared by using glycerol as a plasticizer and citric acid as a crosslinking agent, through solution casting method and their biodegradable properties were studied. The structure of the blend was characterized by Fourier Transform Infrared Spectroscopy (FTIR) and Differential Scanning Calorimeter (DSC). The morphology of starch shows the potato starch has oval or ellipsoid in the structure having a size of 20-35 µm. The compatibility of the two components can be improved by the hydroxyl group present on both starch and PVA. As compared to pure polyvinyl, the melting point of the blend was decreased. The blends were found degradable within a weak.

Keywords: Biodegradable, DSC, starch, polyvinyl alcohol, PVA/starch blend.

Introduction

The plastic we used in our daily life is easy to use, inexpensive, flexible as well as durable. But they are chemically synthesized from petrochemical industries and produced with huge toxic additives. They are non-renewable, non-degradable and lack of infrastructure [1,2]. Being non-degradable they cause white pollution and reduce the resources. The preparation of biodegradable plastic is a wise thought to overcome these problems, which can be prepared from agricultural products [3].

Solanum tuberosum is the third major source of starch [4]. It is naturally occurring agricultural residue and renewable which can be used to produce degradable plastic [5]. Pure starch has low water resistibility, processability, thermal stability, and strength. To overcome these fallacies starch is often blended with other degradable$^{\varepsilon}$polymers such as polylactic acid (PLA), poly--caprolactone (PCL), polyvinyl alcohol (PVA) [2,3,6]. PVA is semicrystalline, non-toxic, water-soluble, having excellent optical and physical properties [7]. The blend of PVA and starch still lacks some mechanical and thermal properties [2]. So, glycerol and citric acid (CA) can be used as a plasticizer and crosslinking agent respectively [8,9].

Several kinds of research have been conducted on the biodegradation of starch, PVA and starch –blends. Starch can be readily metabolized by a range of microorganisms to fermentation products such as ethanol [10,11], hydrogen [12,13] and methane [14]. PVA is also susceptible to biological degradation, however, the process was slow [15]. Furthermore, the overall number of PVA degrading microorganisms was rather limited in comparison to the widespread species able to degrade aliphatic polyesters, such as polyhydroxyalkanoate (PHA) and PLA [16].

Chen *et al*., observed that the rates of biodegradation in starch–PVA cast films were degraded much faster than pure PVA [17]. Mao *et al*. in his work reported that the addition of PVA slowed the degradation process in the samples [9]. Russo *et al.* investigated the degradability of thermoplastic starch and PVA blends under anaerobic conditions and concluded that predominantly PVA remained at the end and that starch was almost entirely degraded [18]. Chai *et al*. evaluated the biodegradability of modified starch–PVA blends with bio-reactivity kinetic models. And conclude, the degradability of PVA was enhanced with the addition of the starch [19]. Cinelli *et al.* characterized the biodegradability with or without a cross-linking agent of PVA, starch, and lignocellulosic films. Results were in favor of biodegradation [20].

Tang; Zou *et al.* done their studied the biodegradability of nano-SiO_2 reinforced starch/PVA nanocomposite films. Results showed that nanoparticles had no significant influence on the biodegradability of films [21]. Popescu *et al.* researched the degradation of clay–starch–PVA nanocomposite films and concluded that the biodegradation of films depended on both type and content of nanoparticles and the nanoparticles hindered the rate of biodegradation [22].

Starch–PVA films can be chosen for the biomedical and clinical field (such as drug control release carrier and biomembrane) [23], while chemically bonded composites can be taken as recyclable sizing agents [24]. PVA/starch has been considered for the replacement of polystyrene foams for expanded foams as loose-fill packaging materials.

According to Vilpoux and Avérous, the commercial alternative to polystyrene (PS) is a blend with 95% of hydroxyl propylated high amylose starch and 5% of PVA [25]. PVA/starch blends are sensitive to moisture but they are good in providing barriers to oxygen and carbon dioxide. So they can act as flavor and aroma barriers, having good resistance to most organic compounds and solvents can be used in food technology [2]. Although there are several kinds of research have done on fabrication and characterization of PVA/starch film with the mechanical test, still there is a lack of research on thermal properties and complete biodegradation. Hence this work is a step further to study thermal behavior and biodegradation of PVA/starch blends.

Materials and Methods

Materials

Solanum tuberosum were collected from National Potato Research Project (NPRP), Khumaltar, Lalitpur in the month of June 2018, they were cultivated in Parwanipur, Bara district and harvested in March and stored in cold store of NPRP, Lalitpur. Polyvinyl alcohol, glycerol, citric acid were brought from Thermo Fisher Scientific India Pvt. Ltd. (India). The water used in the whole thesis work was de-ionized water brought from the local shop of Kathmandu.

Extraction of starch from *Solanum tuberosum*

The process of extraction of starch from *Solanum tuberosum* was done according to Singh and Singh [26]. The tubers of *Solanum tuberosum* was cleaned properly, peeled off and cut into small pieces and were dipped in water containing a small amount of potassium metabisulfite ($K_2S_2O_5$). Then grinned and filtered, the residue was washed with distilled water and left for decantation. After complete decantation, the starch cake was collected and dried at a temperature of 40 ^{0}C in a hot-air cabinet drier.

Preparation of PVA/starch blends

The fabrication of starch with PVA was done by following the protocol mentioned by Shi *et al. via* the solution casting process [23]. The solution was prepared by dissolving the PVA, starch, and glycerol in hot water at 95 ^{0}C to form a homogeneous gel-like solution then citric acid was added into the mixture with a mechanical stirrer for 10 minutes at 30 ^{0}C. Bubbles were removed with an aspirator. The prepared solution was poured on a pre-warmed glass Petri-plate and evaporated from the molds at 25 ^{0}C for one week. The dried films were sealed in polyethylene bags and were stored at 20 ^{0}C to characterize them.

Characterization

Characterization of extracted starch

Moisture content

The determination of moisture content of starch follows the AOAC process [27]. The crucible and lid were dried in the oven at 105 ^{0}C for 3 hours and transferred to desiccator to cool. Weighed it out. Weighed 3 g starch sample was spread uniformly in the crucible. Then the crucible was placed in an oven for 3 hours at 105 ^{0}C. After that, it was transferred to desiccator with the partially covered lid to cool. Reweighted the dish and starch sample. For this, three concurrent data was taken and the mean value was found then moisture content was calculated by the following equation (i).

$$\text{Moisture (\%)} = \frac{(W_1 - W_2)}{W_1} \times 100\ \% \text{ -----------------------(i)}$$

Where, W_1 = weight of sample before drying in gram.

W_2 = weight of sample after drying in gram.

Ash content

To determine the ash content of the starch AOAC method was followed [27]. The crucible and lid were placed in the furnace at 550 ^{0}C overnight. The crucible was cooled in a desiccator for 20 minutes. The crucible and lid were weighed to decimal places. The starch sample was weighed and put into the crucible. The crucible was heated over low Bunsen flame with half covered. When flames were no longer produced, crucible with lid was placed in the furnace. It was heated at 550 ^{0}C overnight. During heating, the crucible was not covered by a lid. The lid was placed on crucible after complete heating, to prevent loss of fluffy ash. Then it was placed in desiccator to cool down. The ash with crucible and lid was weighed. And the ash content was calculated by the following equation (ii).

$$\text{Ash (\%)} = \frac{\text{weight of ash}}{\text{weight of sample}} \times 100\ \% \text{ ----------------------(ii)}$$

Amylose content

A starch sample of 0.10 g was weighed into 100 cm^3 volumetric flask and 1 cm^3 of 99% ethanol and 9 cm^3 of 1 M sodium hydroxide (NaOH) solution were carefully added. The contents were mixed thoroughly and the sample solution was heated for 10 mins in boiling water to gelatinize the starch. After cooling the solution was made up to the mark with distilled water and shaken thoroughly. 5 cm^3 of starch solution in a 100 cm^3 volumetric flask was treated with 1.0 cm^3 of 1M acetic acid and 2.0 cm^3 of iodine solution. The solution was diluted to the mark with distilled water and absorption was read using a spectrophotometer at 620 nm. The absorbance of a blank solution prepared accordingly was subtracted from that of the sample and amylose and amylopectin contents were calculated using equations (iii) respectively.

$$\text{Amylose content (\%)} = 3.06 \times \text{absorbance} \times 20\% \ldots\ldots \text{(iii)}$$

Fourier transform infrared (FTIR) spectroscopy

The FTIR analysis for starch samples was done in the Nepal Academy of Science and Technology (NAST), Khumaltar, Lalitpur by using SHIMADZU spectrophotometer (IR Prestige-21) Nepal. The spectra were obtained at a resolution range of 400 cm^{-1} to 4000 cm^{-1}.

Scanning electron micrograph (SEM)

For surface morphology, starch samples were dehydrated in 99.8% ethanol and sprinkled on carbon tape mounted on an aluminum stub. Then they were coated with a thin gold film using a sputter coater and observed in a FEGSEMMAIA3 (model 2016) microscope (Tescan, Czech Republic) at an accelerating potential of 2 kV.

Characterization of PVA/starch blends

Fourier transform infrared (FTIR) spectroscopy

FTIR analysis for PVA/starch blends was done by IRTracer-100, SHIMADZU in the Central Department of Chemistry, Tribhuvan University, Kritipur, Kathmandu, Nepal. The sample was prepared by mixing the fine powder with KBr and pressing. The spectra were obtained at a resolution range of 400 cm^{-1} to 4000 cm^{-1}.

Differential scanning calorimeter (DSC)

Differential scanning calorimetry thermograms were recorded by NETZSCH DSC 204 F1 phoenix (India) instrument. Sample pieces of about 10 g were placed in an aluminum pan and were first cooled to room temperature and held there for 5 min. After that, a heating scan was conducted from 25 ^{0}C to 250 ^{0}C at a heating rate 10 K/min.

Biodegradation test of PVA/starch blends

Here the process of Azahari *et al.* was performed for the process of biodegradation test of PVA/starch blends [28]. The biodegradation test was performed under compost burial conditions. One vase of the approximate capacity of 5 L was filled with cow's dung compost. The composites were cut into 2 cm × 2 cm in square shape and each sample was enclosed in tea bags individually and made. The samples were buried in the compost at a depth of 10 cm and the moisture of the compost was maintained by sprinkling water at regular intervals of time.

The degradation of the samples was determined at regular intervals of 7 days by carefully removing the sample from the compost and washing it gently with ethanol to remove the trace of compost from the film and dried in an oven at 60 ^{0}C for 4 hours and weight of the samples were taken on a digital balance.

Results and Discussion

Yield of starch

The yield of starch from the tuber of *Solanum tuberosum* was calculated using the standard equation, on the basis of the mass of raw *Solanum tuberosum* sample of starting material. Using raw *Solanum tuberosum* sample as reference the yield of starch was found to be 3.82 % in Khumal Rato (KR).

The yield of starch was found affected by various artificial factors including wet storage [29], genetic variation [30,31], the drying conditions of the raw materials (oven-dried chips and flour have higher yield than sun-dried ones) [32], harvesting time and root storage (root storage decreased starch content) [33,34], the form of the roots used for extraction (fresh root gave better extraction efficiency and purer starch than dried chips and pellets) [35], and the environmental conditions for growing the crop (e.g., two growth locations gave significant difference in the starch yield of the same variety) [30]. Thus for high starch yield, varieties with high dry matter content and thin peel thickness are suitable [36]. All these factors can be controlled, and careful selection of the parameters would lead to a maximum of starch yield. It should be noted that high yield does not necessarily mean the starch would have the desired quality for any specific application.

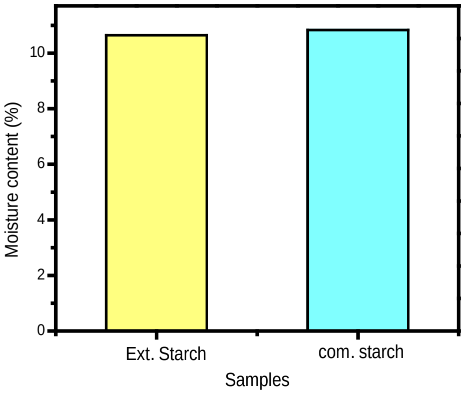

***Figure 1:** Moisture content of extracted starch and commercial starch.*

The moisture content of extracted starch from Khumal Rato and commercial starch is represented in Figure 1.

The result showed that the moisture content of Khumal Rato was 10.65% and commercial starch was 10.84%. The relatively low moisture content of the starches makes them easy to store at room temperature and less prone to colonization by organism degradation as shown for root, tuber and cereal starches [37,38] making them suitable for utilization in industries like the filler in biodegradable plastic and pharmaceutical industry that make use of low moisture content. The range of moisture contents in these starches varied from 7.54 to 9.37%. Tapioca starch showed slightly lower moisture content (7.54%) than corn starch (7.74%) and similar to the results of on mango starch [39]. Potato starch had a significantly higher moisture level (9.37%) than other starches.

Ash content of extracted starch and commercial starch is shown in Figure 2. From the graph, the ash content of the starch from the Khumal Rato was 0.7% and commercial starch was 0.8. However, the ash content of the potato was significantly higher than 0.20 to 0.22% for Oat starches [40]. The ash content is an estimate of the total mineral content of the starch. The low ash content is an indication of the good quality of the starches, because high mineral content is sometimes used to retard the growth of certain microorganisms [36].

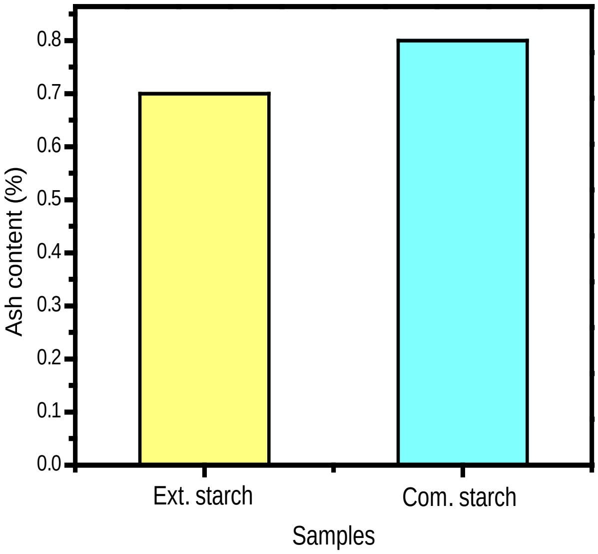

***Figure 2:** Ash content of extracted starch and commercial starch.*

The amylose content of starch determines its properties (such as water-binding capacity, thickening, gelling, etc.) and as a result dictates most of its uses. The results obtained showed significant variations. The amylose contents (in terms of amylose equivalent) for extracted starches from Khumal Rato was found to be 23.26%, higher than values reported for mango [39] and corn starches 16.9–25.3 % [41] and also higher than that of oat [40] and of tapioca (16.27%). These values were slightly higher than those reported by Glicksman and Rapaille and Vanhemelrijck for potato (22%) starch, but comparable to that for corn (26%) and tapioca (17%) starches. Thus, the starch could be applied in industries that produce, fillers, thickeners, binders, etc.[42].

Xia *et al.,* reported that the structure of amylose is sensitive to physical and chemical treatments. When slight applied of heat change the amount of content.

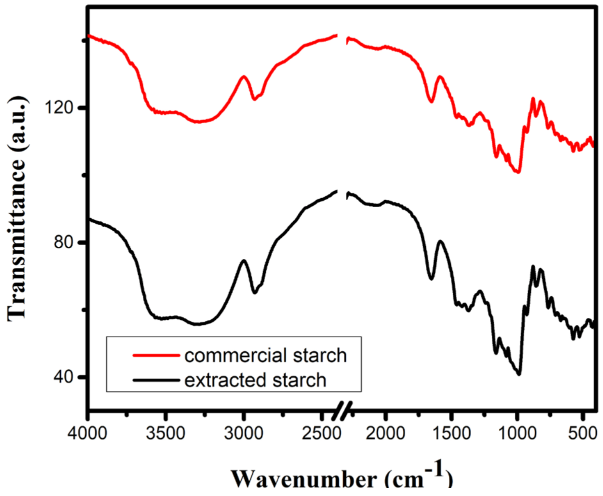

Figure 3: *FTIR spectra of extracted starch and commercial starch.*

The FTIR spectra of extracted starch and commercial starch are presented in Figure 3, whereas the interpretation of each peak is given in Table 1.

As shown in the graph, the presence of an absorption band at around 3600-3300 cm^{-1} for both starch is due to the vibration of the hydroxyl groups (-OH). The band at 2927 cm^{-1} is the characteristic peak of the C-H stretching vibration of the amylase and amylopectin, present in starch. The peak occurred for extracted starch at 1645 cm^{-1} is due to the tightly bound water molecules. The changes in the crystallinity of the starch are reflected around this band. The band at 1157 cm^{-1} of extracted starch and commercial starch is assigned as a complex mode involving the CH_2OH side chain in amylase.

In addition, the characteristic C-O-C ring vibration on starch leads to an absorbance peak at around 700-900 cm^{-1}. The C-O bending associated with the OH group would cause an absorbance peak at around 1648 cm^{-1}. Furthermore, the absorbance peak at 1419 cm^{-1} implied the presence of C-H symmetrical scissoring of CH_2OH moiety. The uncommon CO_2 peak in starch (2358 cm^{-1}) was observed in the potato starch IR spectrum. It might result from measuring conditions [43].

SEM is used to investigate the granule morphology of the potato starch. The morphology of starch was shown in Figures 4 and 5.

Table 1: *Band assignment of potato starch.*

S. No.	Functional group	Wave number cm^{-1}	Potato starch cm^{-1}
1	O-H stretching	3600 – 3300	3569-3170
2	C-H stretching	2931	2931
3	C-O bending associated with OH group	1637	1645
4	CH_2 symmetric deformation	1458	1437
5	CH_2 symmetric scissoring	1415	1419
6	C-H symmetric bending	1385-1375	1381
7	C-O-C asymmetric stretching	1149	1157
8	C-O stretching	1200-800	993
9	C-O-C ring vibration of carbohydrate	920,856; 758	929, 858,763

The starch image has 20 and 50 resolutions. The image revealed that the potato starch granules exits in smooth granule form have oval and ellipsoid shapes with sizes ranging from 5 to 35 µm in width and 15 to 60 µm in height.

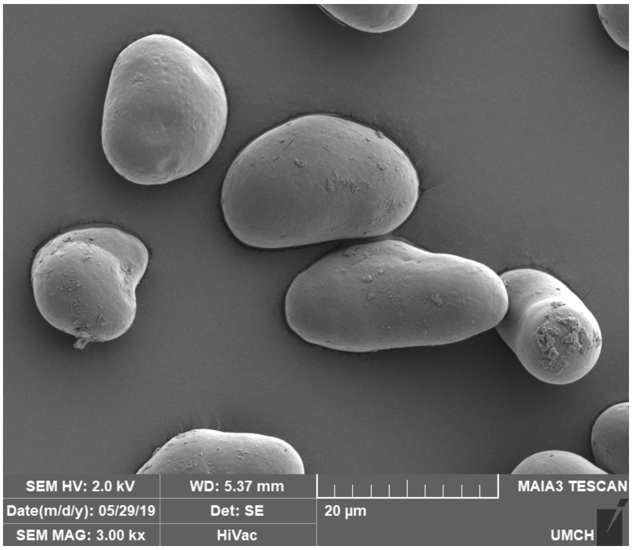

Figure 4: *SEM image of potato starch having 20 µm resolution.*

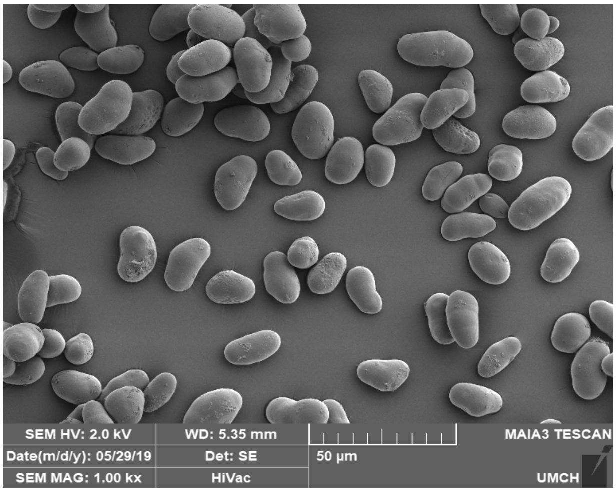

Figure 5: *SEM image of the potato starch powder having 50 µm resolution.*

Wang reported in his work, the sizes of small and large granules ranged from 0.4 to 3µm and from 6 to 40 µm in maize starch, from 0.6 µm and from 10 to100 µm in potato starch, and from 0.6 to 6µm and from 10 to 70 µm in pea starch, respectively. Fannon et al. mentioned that SEM has been used to relate granule morphology to starch genotype. Svegmark and Hermanson reported that the variation in size and shape of starch granules is due to the biological origin. Singh and Singh claimed that the morphology of granules depends on the biochemistry of chloroplast or amyloplast and physiology of plants [44].

Characterization of PVA/starch blends

The fabricated starch/PVA blends were transparent, flexible, are characterized via FTIR, DSC and biodegradation test.

The FTIR spectra of PVA/starch blends with different content of PVA shown in Figure 6 and band assignment is shown in Table 2.

Broad and strong absorption band from 3200-3600 cm^{-1} is the cause of the stretching vibration of -OH present in starch. The absorption band 2931 cm^{-1} is due to an aliphatic saturated C-H stretching vibration of starch.

Another absorption band at 1640 cm^{-1} is because of the stretching vibration of C=O and at 1651cm^{-1} is due to the bending mode of the absorbed water and some contribution from the carboxylate group. The vibration of the CH_2 group produces an absorption band at 1409 cm^{-1}. Due to the cause of C-O stretching an absorption band at 1035 cm^{-1} was produced.

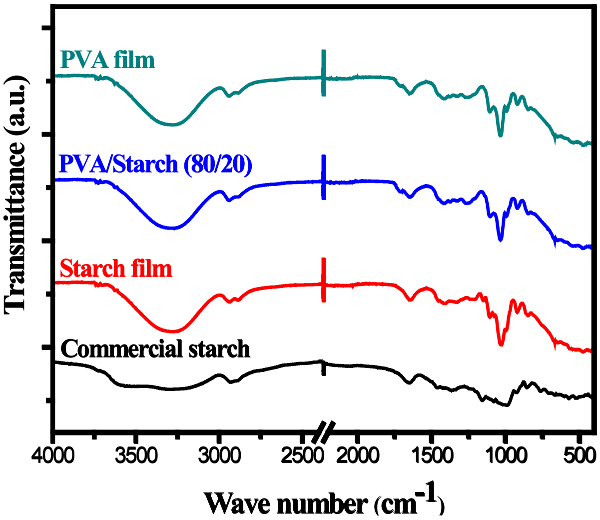

Figure 6: *FTIR spectra of commercial starch and PVA/ starch blends*

Blout *et al.,* reported that a peak at 2361 cm^{-1} is due to atmospheric carbon dioxide. Peaks due to water in the films occur at 1652 cm^{-1} and around 3240 cm^{-1}. The later peak overlaps the peak due to the -OH stretching of hydroxyl groups.

Table 2: *Characteristics FTIR bands starch/PVA blend.*

S.N.	Functional group [3,8]	Wave number (cm^{-1}) [3,8]	Blends
1	-OH stretching	3600-3200	3285
2	C-H	3000-2850	2931
5	C-C	1636	1640
6	CH_2	1461-1417	1409
8	C-O	1141-1093	1035
9	C-O-H	669	652

Another research by Finch *et al.*, mention that peaks at 2912, 1324 and 843 and 1084 cm^{-1} are attributed to the C–H stretching, C–H bending and C–O stretching of PVA, respectively. The broad high absorption peak at 3237 cm^{-1} is assumed to arise from the -OH stretching frequencies of PVA and water hydroxyl groups. The band at 1708 cm^{-1} was attributed to the carbonyl functional groups due to residual acetate groups remaining after the manufacture of PVA from hydrolysis of polyvinyl acetate or oxidation during manufacturing and processing [44].

Differential Scanning Calorimeter (DSC) provides information about thermal changes that do not involve a change in sample mass change in sample mass. Now it is a more commonly used technique than TGA. The results of the thermal behavior of PVA and starch with glycerol and citric acid, was shown in Figure 7. From the DSC curve, the melting point (T_m) of PVA blend without starch was found 198 ^{0}C, and the melting point for PVA/S 5%, PVA/S 20%, PVA/S 40% were found 141 ^{0}C , 137 ^{0}C and 131 ^{0}C respectively.

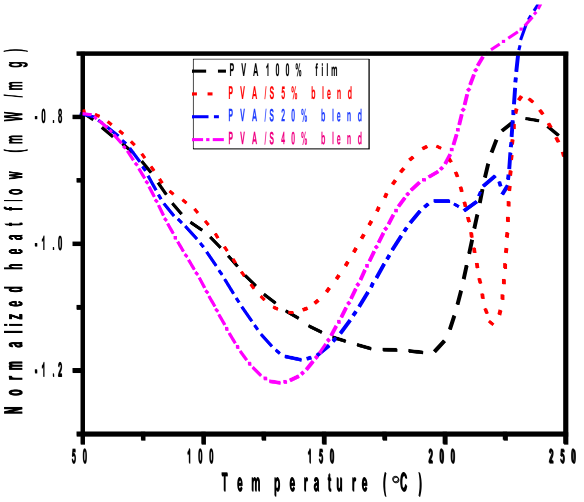

Figure 7: *DSC curve of PVA/starch blends with glycerol and citric acid.*

The observed T_m is lower than that of reported one which is 230 ^{0}C for pure PVA. This is because the PVA film had already contained some water as the plasticizer. When glycerol was added, the T_m decreased. The reduction in T_m was attributed to the reduction in the cohesive forces of attraction between the polymer chains. The plasticizer molecules penetrated the polymer matrix because they were smaller than the polymer molecule. As a result, polar attractive forces were established between the plasticizer and chain segments, which were responsible for the reduction of the cohesive forces and, therefore, a reduction of T_m. Shi *et al.* and Shreedhar *et al.* also reported the decreased of T_m in their work [23].

The biodegradation result showed that the buried blends were found completely degraded after taken out from the vase. So the calculation of weight loss couldn't perform. There is the various possible reason behind the degradation. Lucas *et al.* mentioned that in the process of biodegradation, the resistance and durability of polymeric materials are made weak by the action of microorganisms or/and environmental abiotic factors. Another suitable possible reason was given by Bonhomme *et al.* and Leja *et al.* according to them, the growth of microorganisms adhering to the polymer surface can provoke the formation of cracks, increase the size of pores and others defects, that may lead to the disintegration of a material into small fragments [45].

Conclusion

Starch was successfully extracted from locally cultivated tubers' potatoes (*Solanum tuberosum*). It is white, an amorphous in nature, tasteless, odorless, insoluble in cold water, alcohol, and ether. The preliminary results indicate the quality of extracted starch is good and they are as reliable as commercial starch. The FTIR peak at 2919 cm^{-1} indicates the presence of amylose and amylopectin. Morphology of starch shows, smooth granule, oval and ellipsoid in shape of 5-35 µm in size.

The film of starch and PVA was successfully fabricated by solution casting methods with the use of glycerol as a plasticizer and citric acid as the crosslinking agent. The fabricated blends were transparent, flexible and smooth. The blend was physically bonded. The plasticizer and crosslinking agent help blend to increase flexibility. The physically bonded blend is easily affected by moisture, temperature and air. -OH groups on starch and PVA formed hydrogen bonding interactions, which could improve the compatibility of the two components. As compared to pure polyvinyl, the melting point of the blend was decreased and the blend was found degradable in nature.

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