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OBTAINING COMPOSITE MATERIALS BASED ON POLYPROPYLENE AND STARCH

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ABSTRACT

Due to the large-scale utilization of plastic products in our lives and their long decomposition time, nature is becoming disposed of with plastic waste every year. In this work, the opportunity of obtaining biodegradable compositions based on synthetic and natural polymers for single-use plastic products is re-searched. The opportunity of obtaining thermoplastic starch was researched and the structure and properties were studied, and a composition based on thermo-plastic starch with polypropylene was obtained. FTIR-spectroscopy and radio-graphic methods were used to determine the optimal conditions of thermoplastic starch production. The amorphous-crystalline structure and mechanical proper-ties of the composites based on thermoplastic starch and polypropylene were studied. The composition obtained based on research has the property of biodeg-radability and it can be used in the production of single-use plastic products for food.

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Introduction

These days, synthetic polymers are widely used in different areas of human life. 36% of manufactured plastics are utilized as packaging materials. As the population of the world grows, the necessity for food also increases, which leads to an increase in the demand for the utilization of food packaging materials for storage and transportation. The recycling of single-use packaging for food is not economically efficient [1]. One of the ways to reduce the problem of plastic waste in nature is the use of biodegradable raw materials in the production of disposa-ble packaging. The utilization of starch, which is relatively common in replacing polyolefin polymers with biodegradable analogs, is technically and economically preferable to other biodegradable natural polymers [2]. Most of the waste collect-ed in landfills is synthetic polymer products used as packaging. Non-biodegradable synthetic polymers accumulate and create a plastic waste problem in the environment. Starch, biodegradable polymer that is biosynthesis by many plants, is one of the most abundant renewable resources known to man[3]. Bio-degradable polysaccharides utilized in composites with synthetic polymers, in particular starch, are not included in thermoplastic polymers. Plasticizers are used to make starch and other polysaccharides thermoplastic. [4]. It gives an op-portunity that obtains compositions with polyolefins containing a high amount of thermoplastic starch and uses them in the production of disposable packaging materials on a large scale, reducing the harmful impact of waste on nature. [5,6]. According to the literature, replacing one ton of plastic with biopolymers reduces the amount of CO2 released into nature by 3.2 tons [7].

The purpose of the work is to research the properties of composite materials based on corn starch and polypropylene for thermoplasticization of local corn starch and use in the production of biodegradable disposable packaging materi-als. Would produce two-phase blends due to a large incompatibility between nonpolar PP and highly polar TPS, hence PP and TPS are not uniform distribu-tion miscible. Biodegradation of maleated linear low-density polyethylene and starch blends. Polymer degradation and stability. Compatibilizers are used to in-crease the compatibility of non-polar and polar and to obtain an evenly distributed composition.

Experimental

As materials for the research, J-320 brand polypropylene (PP), Maleinated polypropylene (PP-MAN), corn starch (Starch) for food produced at Russia's "AMILCO" LLC, glycerol (propane-1,2,3-triol) and sorbitol ((2S,3R,4R,5R)-Hexane-1,2,3,4,5,6-hexol) as plasticizers, was selected.

The starch was dried at 80°C for 12 hours and the gelatinization process was carried out in a single-screw laboratory extruder under the influence of plasticizers: glycerol and sorbitols at a temperature of 105-115-125°C, at a speed of 50 revolutions/min, and samples of thermoplastic starch (TPS) were taken (Table 1)

Table 1

The amount of plasticizers in the gelatinization process

Sample code	Total plasticizer content (wt%)	Plasticizer ratios (glcerol-wt/ sorbitol-wt)
$TPS_{10/00}$	10	1:0
$TPS_{20/00}$	20	2:0
$TPS_{30/00}$	30	3:0
$TPS_{13/26}$	39	1:2
$TPS_{26/13}$	39	2:1

Wide-angle X-ray diffraction was carried out with an XRD “MiniFlex 600” (Rigaku, Japan) in reflection mode. Cu–Kα radiation was employed as a radiation source. The scanning range was 5° to 48° at a rate of 2°/min. The generator was operated at 22 kV and 12 mA. Calculations of the structural parameters of the samples were performed using SmartLab Studio II software for MiniFlex (Version 4.3.200.0, Japan).

The structure of the samples was investigated in the IK-spectrophotometer “Inventio-S” (Bruker, Germany) in the range from 500 cm-1 to 4000 cm-1. Composition samples were taken based on TPS and PP obtained under the optimal conditions selected in a single-screw extruder at a temperature of sections of 160-165-175°C, a speed of 50 rpm. It was used as a compatibilizer in maleinated polypropylene to improve the distribution of components depending on the amount of TPS in the composition [8].

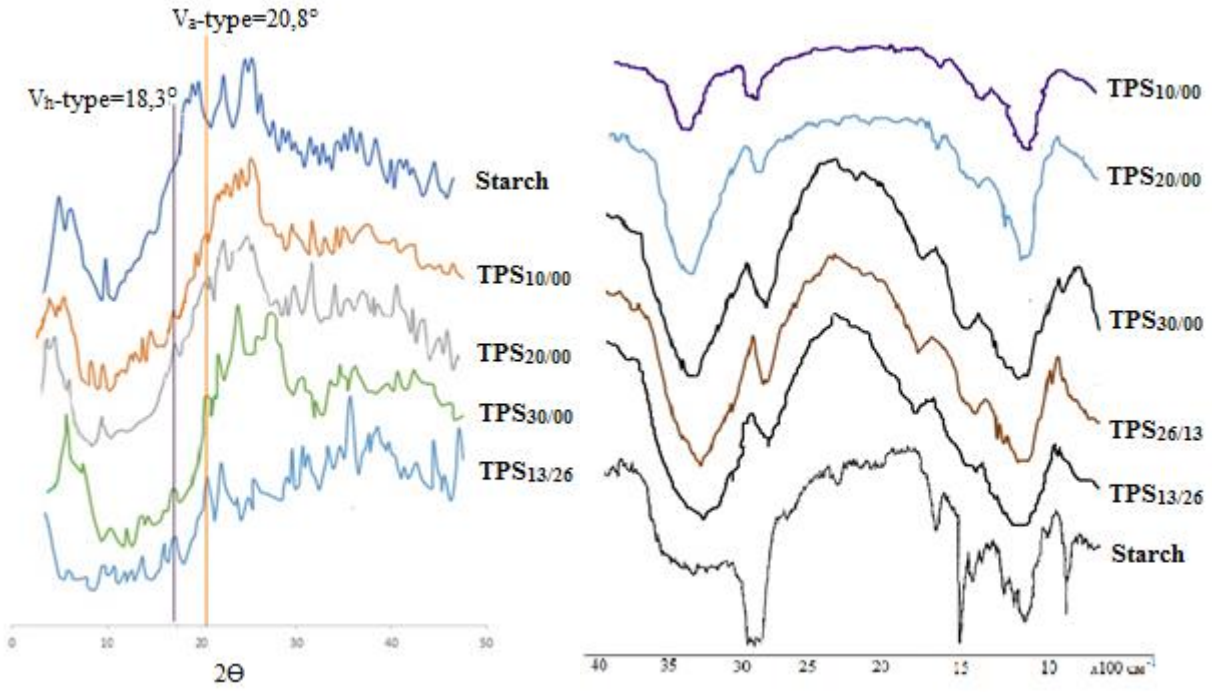
Measurements of the mechanical properties (the modulus of young, tensile strength (σ) and elongation (ε)) of the composites were performed using an AG-X plus tensile tester (Shimadzu, Japan) according to standard methods at a 10 mm/min crosshead speed with ASTM D638 “Method of testing elasticity” [9].

The thermal analysis of samples was recorded with an STA TG-DTA/DSC “Start-1600” (Linseis, Germany) by heating ~20 mg of samples at 10 °C/min under an air atmosphere from ambient temperature to 700°C.

Results and Discussion

The structural evolution of thermoplastic starch (TPK) systems plasticized with different polyol formulations was systematically investigated using X-ray diffraction (XRD) and Fourier-transform infrared (FTIR) spectroscopy. The combined application of these techniques provides

complementary information on both the crystalline organization and the molecular-level interactions occurring during starch gelatinization .



a b

Figure-1. *X-ray diffractogram (a) and IR spectra of TPS samples (b)*

XRD diffractograms of native starch and gelatinized TPK samples are pre-sented in Figure 1a. Native starch exhibits characteristic reflections associated with its semicrystalline granular structure. Following gelatinization, a pro-nounced disruption of the native crystalline lattice is observed, evidenced by the reduction in overall crystallinity and the attenuation of native starch reflections. Concomitantly, new diffraction peaks appear at $2\theta \approx 18.3°$ and $2\theta \approx 20.8°$, which are assigned to Vh-type and Va-type crystalline polymorphs, respectively. These V-type structures are not present in native starch and are generally attributed to the formation of amylose–plasticizer inclusion complexes, where amylose helices reorganize to accommodate low-molecular-weight polyols within their cavities.

The emergence of Vh and Va crystalline phases indicates a substantial rear-rangement of starch macromolecules during thermoplastic processing. Such structural transformation is consistent with previously reported gelatinization mechanisms and confirms literature observations [10]. Moreover, the intensity of V-type diffraction peaks increases with increasing plasticizer content, suggesting enhanced chain mobility and more effective reorganization of starch helices into thermodynamically favored crystalline domains. Among the investigated compo-sitions, TPS30/00 and TPS13/26 exhibit the highest diffraction intensities, indi-cating the most pronounced development of V-type crystallinity.

To elucidate the intermolecular interactions governing these structural changes, FTIR spectroscopy was employed (Figure 1b). The FTIR spectra of TPS samples display a broad absorption band centered around 3400 cm^{-1}, corre-sponding to O–H stretching vibrations associated with intermolecular hydrogen bonding. Compared with native starch, gelatinized samples exhibit a marked in-crease in the intensity and broadening of this band, reflecting a significant en-hancement in hydrogen bond density within the TPS matrix.

A progressive increase in the 3400 cm^{-1} band intensity is observed with in-creasing plasticizer concentration, indicating that polyol plasticizers actively par-ticipate in hydrogen bond

formation with starch hydroxyl groups. The presence of multiple –OH functionalities in glycerol and sorbitol facilitates the formation of extensive hydrogen-bonding networks, thereby strengthening intermolecular attractions between starch macromolecules and plasticizer molecules. Notably, TPS30/00 and TPS13/26 demonstrate the highest intensities in the hydrogen-bonding region, in direct agreement with the XRD results showing maximal V-type crystalline phase formation.

The strong correlation between XRD and FTIR data confirms that hydrogen bonding plays a critical role in stabilizing Vh- and Va-type crystalline structures. Enhanced hydrogen-bond interactions promote the formation and stabilization of amylose–plasticizer inclusion complexes, which act as structural motifs for V-type crystallization. Based on the combined structural and spectroscopic anal-yses, TPS30/00 (containing 30 wt% glycerol) and TPS13/26 (containing 13 wt% glycerol and 26 wt% sorbitol) were identified as optimal formulations for ther-moplastic starch production.

Table 2

Component ratios of composition samples

Sample code	PP (wt,%)	PP-MAH (wt,%)	$TPS_{13/26}$ (wt,%)
^{100}PP (J-520)	100	-	-
^{67}PP/^{3}PP-MAH/^{30}$TPS_{13/26}$	67	3	30
^{45}PP/^{5}PP-MAH/^{50}$TPS_{13/26}$	45	5	50
^{23}PP/^{7}PP-MAH/^{70}$TPS_{13/26}$	23	7	70

The crystalline characteristics of neat polypropylene (PP) and PP/PP-g-MAH/TPS composites containing various amounts of thermoplastic starch were examined using X-ray diffraction (XRD). The resulting diffractograms are shown in Figure 2. This analysis was carried out to assess the influence of TPS addition and reactive compatibilization on the crystalline arrangement of the PP matrix.

The XRD pattern of neat polypropylene (100PP) displays distinct diffrac-tion peaks at 2θ values of approximately 14.1°, 16.9°, 18.6°, 21.2°, and 25.5°, which are typical of the α-crystalline phase of isotactic polypropylene. The pro-nounced intensity and sharp nature of these reflections indicate a highly ordered crystalline structure and a high crystallinity level in the unfilled PP sample.

After incorporating thermoplastic starch and PP-g-MAH, the composite dif-fractograms continue to exhibit the characteristic α-PP reflections, confirming that the fundamental crystalline structure of polypropylene is retained across all formulations. Nevertheless, an incremental reduction in peak intensity accompa-nied by slight peak broadening is observed as the TPS content increases. This trend suggests a gradual decrease in the crystallinity of the PP phase and a reduc-tion in crystalline perfection.

In composites with moderate TPS contents (67PP/3PP-g-MAH/30$TPS_{13/26}$ and 45PP/5PP-g-MAH/50$TPS_{13/26}$), the crystalline reflections of PP remain clearly distinguishable, though with lower intensities compared to neat PP. This reduc-tion is attributed to constrained mobility of PP molecular chains caused by the presence of finely dispersed TPS domains, along with enhanced interfacial inter-actions introduced by PP-g-MAH. The compatibilizer improves the dispersion of TPS within the PP matrix, thereby hindering lamellar crystal growth and leading to the formation of smaller or less perfect PP crystallites.

At a higher TPS loading (23PP/7PP-g-MAH/70$TPS_{13/26}$), a more substantial attenuation of diffraction peak intensities is evident. This behavior indicates a significant decrease in PP crystallinity, which can be ascribed to the increasing dominance of the TPS phase and the partial loss of continuity in the PP matrix. Under these conditions, restricted chain folding and crystallization result in a structure with a higher amorphous fraction

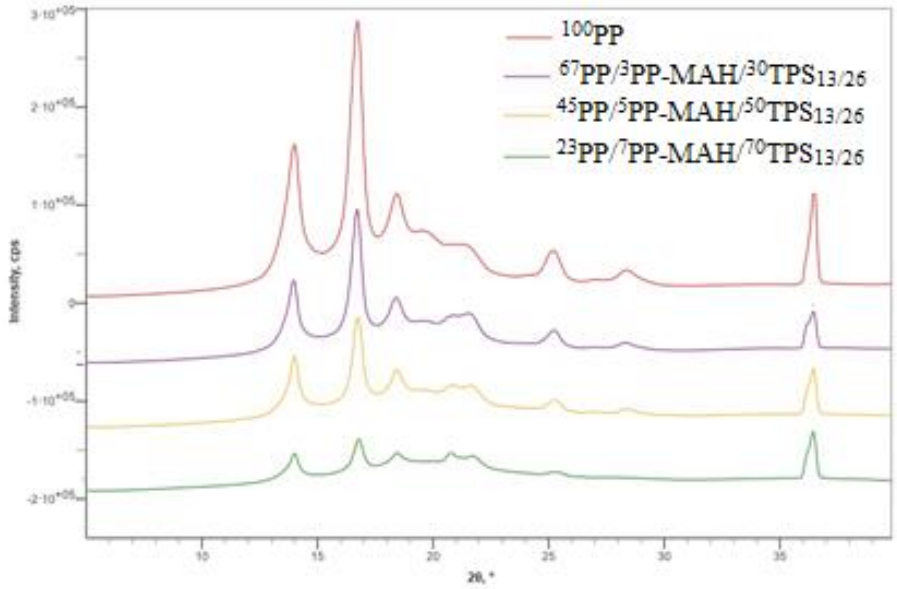


Figure 2. *X-ray diffractograms of composite materials*

The mechanical properties of neat polypropylene (PP) and PP/PP-g-MAH/TPS composites with different thermoplastic starch contents are summa-rized in Table I. The results clearly demonstrate that the incorporation of TPS significantly influences the stiffness, strength, and deformation behavior of the composites.

PP (J-320) exhibits a Young's modulus of 678 MPa, tensile strength of 26.0 MPa, and a high elongation at break of 995%, confirming its ductile nature and continuous polymer matrix structure. Upon incorporation of TPS, a systematic reduction in tensile strength and elongation at break is observed, accompanied by a pronounced increase in material stiffness.

As the TPS content increases from 30 to 70 wt%, the tensile strength de-creases from 20.2 MPa (67PP/3PP-g-MAH/30$TPS_{13/26}$) to 9.6 MPa (23PP/7PP-g-MAH/70$TPS_{13/26}$), while the elongation at break sharply declines from 590% to only 53%. This behavior indicates a progressive loss of ductility and load-bearing capability with increasing TPS concentration.

The reduction in tensile strength and elongation at break can be attributed primarily to two factors. First, despite the presence of PP-g-MAH as a compati-bilizer, the interfacial adhesion between the hydrophilic TPS phase and the hy-drophobic PP matrix remains limited. As TPS content increases, the interfacial area between the dispersed TPS domains and the PP matrix expands, promoting stress concentration and premature failure under tensile loading. Second, the in-creasing volume fraction of TPS reduces the effective load-bearing cross-section of the continuous PP matrix, leading to higher localized stresses during defor-mation.

This trend is consistent with classical composite theory for thermoplastics filled with rigid or semi-rigid natural fillers, where increasing filler content typi-cally results in a monotonic decrease in tensile strength and elongation at break due to insufficient stress transfer across the filler–matrix interface. The observed mechanical behavior suggests that TPS domains act predominantly as stress-concentrating inclusions rather than reinforcing elements under tensile loading.

In contrast to tensile strength and elongation, Young's modulus shows a clear decreasing trend with increasing TPS content, dropping from 518 MPa to 215 MPa as TPS loading increases from 30 to 70 wt%. This reduction indicates that, at higher TPS contents, the plasticized starch phase dominates the mechani-cal response of the composite. The inherently lower stiffness of $TPS_{13/26}$ com-pared to neat PP, combined with increased plasticizer content, results in a softer and more compliant material.

At moderate TPS content (30–50 wt%), the composites retain a balanced mechanical profile, where sufficient stiffness is maintained while avoiding exces-sive brittleness. However, at high TPS loading (70 wt%), the composite structure becomes discontinuous, leading to severe deterioration of tensile properties and a transition toward brittle failure behavior (Table 2).

Table 2

Mechanical properties of compositions

Sample code	Young's modulus, MPa	Tensile strength (σ), MPa	Elongation (l), %
PP (J-320)	678	26,0	995
^{67}PP/^{3}PP-MAH/^{30}TPS$_{13/26}$	518	20,2	590
^{45}PP/^{5}PP-MAH/^{50}TPS$_{13/26}$	401	14,8	426
^{23}PP/^{7}PP-MAH/^{70}TPS$_{13/26}$	215	9,6	53

The interfacial chemical structure of polypropylene (PP)/thermoplastic starch (TPS) composites compatibilized with maleinated polypropylene (PP-g-MAH) was investigated using Fourier-transform infrared (FTIR) spectrosco-py(Figure 3). FTIR analysis was employed to elucidate the nature of molecular interactions and possible chemical reactions occurring between the composite constituents during melt processing.

The FTIR spectrum of neat PP is dominated by characteristic absorption bands corresponding to aliphatic C–H stretching vibrations at 2950–2850 cm^{-1} and deformation vibrations at approximately 1450 and 1375 cm^{-1}, reflecting the nonpolar hydrocarbon structure of the polymer backbone. Upon incorporation of TPS and PP-g-MAH, additional absorption bands appear, indicating significant chemical and structural modifications within the composite system.

A prominent new absorption band is observed at 1721 cm^{-1}, which is at-tributed to the stretching vibration of carbonyl (C=O) groups. The emergence of this band provides direct evidence for the occurrence of a chemical esterification (etherification) reaction between the anhydride functionalities of PP-g-MAH and the hydroxyl (–OH) groups of thermoplastic starch. This reaction leads to the formation of covalent ester linkages at the PP/TPS interface, confirming that PP-g-MAH acts as a reactive compatibilizer rather than a purely physical blending agent.

With increasing PP-g-MAH and TPS content in the composite, the intensity of the 1721 cm^{-1} absorption band increases progressively. This trend indicates a higher extent of interfacial chemical bonding due to the increased availability of reactive anhydride and hydroxyl groups. The growth of this peak suggests that the degree of esterification is composition-dependent and is enhanced under melt-processing conditions, where elevated temperature and shear promote interfacial reactions.

The formation of ester bonds at the interface significantly improves compat-ibility between the hydrophobic PP matrix and the hydrophilic TPS phase. Through chemical coupling, the interfacial tension is reduced, leading to a more uniform dispersion of TPS within the PP matrix and suppression of macroscopic phase separation. This improved interfacial adhesion facilitates more efficient stress transfer across the phase boundary, which is essential for achieving struc-turally stable polymer composites.

Similar esterification reactions between maleic anhydride-grafted polyolefins and starch-based materials have been reported by other researchers, confirming that this mechanism is characteristic of compatibilized PP/starch systems. The present FTIR results are therefore consistent with established reaction pathways and further substantiate the role of PP-g-MAH as an effective chemical bridge be-tween PP and TPS phases.

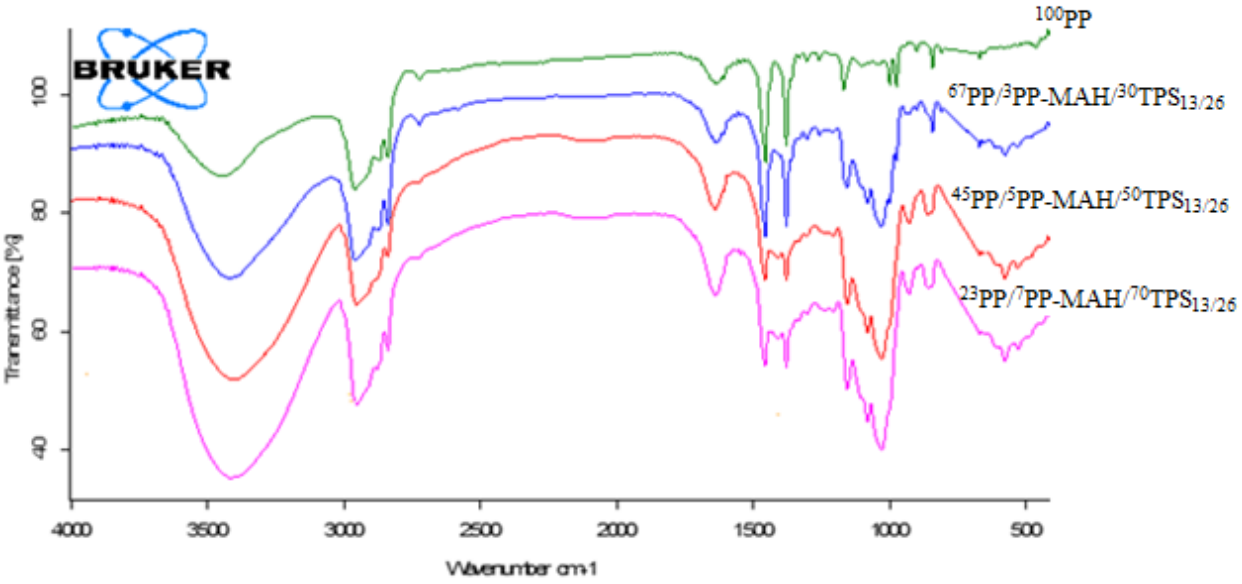


***Figure 3**. FTIR spectra of PP and PP/TPS composite*

The thermal stability and degradation behavior of neat polypropylene (PP) and PP/PP-g-MAH/TPS composites were investigated by thermogravimetric analysis (TGA), and the corresponding mass loss curves are presented in Figure 4.

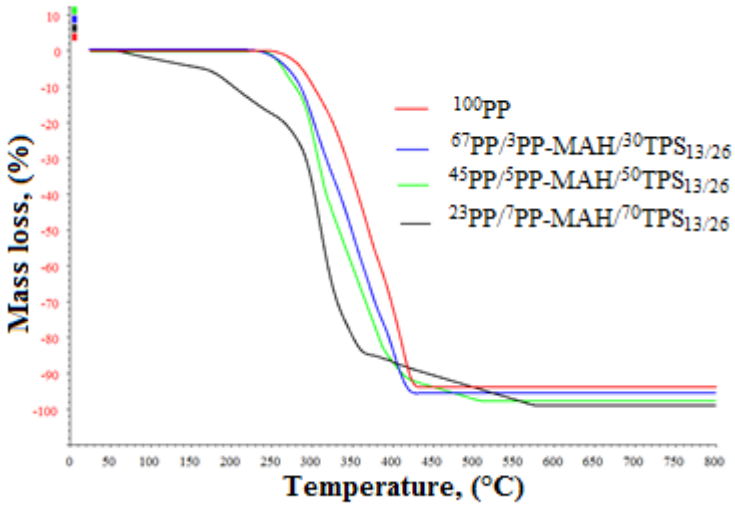


***Figure 4.** TGA of of PP and PP/TPS composite*

The analysis allows for a comparative evaluation of the influence of ther-moplastic starch (TPS) content and compatibilization on the thermal resistance of the composite systems. Neat PP (100PP) exhibits a single-step thermal degrada-tion profile, characteristic of polyolefins, with negligible mass loss below approx-imately 300 °C. The main degradation stage occurs in the temperature range of ~350–450 °C and is associated with random chain scission and depolymerization of the polypropylene backbone. This behavior confirms the high thermal stability of pure PP under inert conditions. In contrast, PP/PP-g-MAH/TPS composites display a more complex degradation behavior, reflecting the multicomponent na-ture of the system. The onset of mass loss for the composites shifts to lower temperatures with increasing TPS content, indicating a

reduction in thermal sta-bility relative to neat PP. This early-stage mass loss is primarily attributed to the thermal decomposition of the TPS phase, which includes dehydration, depoly-merization of starch macromolecules, and degradation of plasticizers present in the thermoplastic starch.

For composites containing lower TPS content (67PP/3PP-g-MAH/30$TPS_{13/26}$ and 45PP/5PP-g-MAH/50$TPS_{13/26}$), the degradation curves still exhibit a domi-nant degradation step associated with the PP matrix, although the onset tempera-ture is slightly reduced compared to neat PP. This behavior suggests that, despite the presence of TPS, the PP matrix remains the primary load-bearing and ther-mally stabilizing phase, particularly at moderate TPS concentrations.

As the TPS content increases to 70 wt% (23PP/7PP-g-MAH/70$TPS_{13/26}$), a pronounced shift of the degradation curve toward lower temperatures is ob-served. This sample shows significant mass loss beginning at substantially lower temperatures, indicating that the TPS-rich phase governs the thermal degradation process. The enhanced contribution of TPS leads to a reduction in the overall thermal resistance of the composite due to the inherently lower thermal stability of starch-based materials compared to polypropylene.The final residual mass at elevated temperatures (above ~500 °C) is slightly higher for TPS-containing composites than for neat PP. This behavior can be attributed to the formation of carbonaceous char from starch decomposition, which is typical for polysaccha-ride-based materials. The presence of such char residues indicates that TPS un-dergoes dehydration and carbonization reactions, contributing to the residual mass after complete polymer degradation.It is important to note that the incorpo-ration of PP-g-MAH as a compatibilizer mitigates, but does not completely elimi-nate, the reduction in thermal stability caused by TPS addition. The esterification reactions between PP-g-MAH and TPS, as confirmed by FTIR analysis, improve interfacial adhesion and promote a more homogeneous phase distribution. This enhanced compatibility contributes to a more gradual degradation process, par-ticularly at intermediate TPS contents, by limiting premature phase separation and localized thermal degradation.

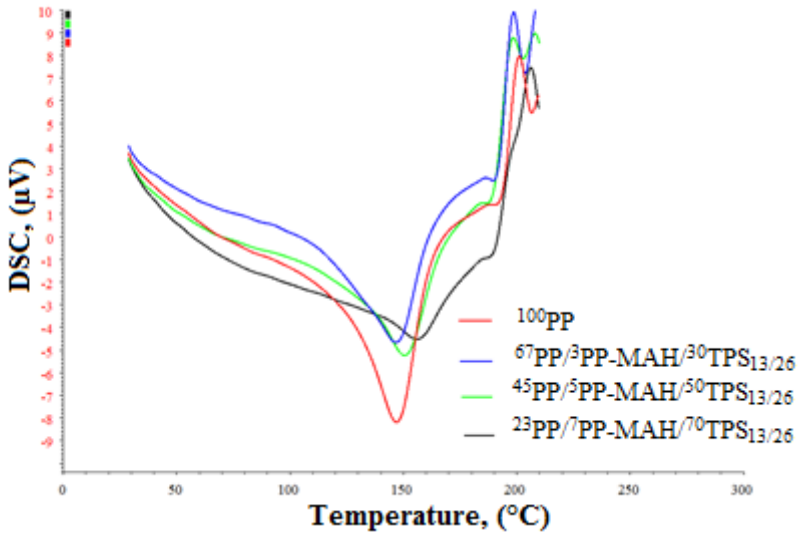


Figure 5. *DSC curves of PP and samples PP/TPS composite*

The thermal transition behavior of neat polypropylene (PP) and PP/PP-g-MAH/TPS composites was investigated by differential scanning calorimetry (DSC), and the corresponding thermograms are presented in Figure 5. DSC anal-ysis provides insight into the melting, crystallization, and phase interaction be-havior of the composite systems, which is essential for understanding their ther-mal and structural stability. PP (100PP) exhibits a well-defined endothermic melt-ing peak centered around 160–165 °C, corresponding to the melting of α-crystalline polypropylene domains. The sharpness and intensity of this peak in-dicate a relatively

high degree of crystallinity and a uniform crystalline structure within the PP matrix. Upon heating, an exothermic crystallization-related transi-tion is also observed at higher temperatures during cooling, reflecting the recrys-tallization behavior of PP.

Incorporation of thermoplastic starch (TPS) and PP-g-MAH into the PP ma-trix leads to noticeable changes in the DSC profiles. For all composite samples, the PP melting endotherm remains observable, indicating that the crystalline phase of PP is preserved despite the presence of TPS. However, a gradual shift of the melting peak toward lower temperatures and a reduction in peak intensity are observed with increasing TPS content. This behavior suggests a decrease in PP crystallinity due to restricted chain mobility and disrupted crystal growth caused by the dispersed TPS phase.

At moderate TPS loadings (67PP/3PP-g-MAH/30$TPS_{13/26}$ and 45PP/5PP-g-MAH/50$TPS_{13/26}$), the melting behavior of PP remains relatively well-defined, alt-hough the endothermic peak becomes broader. The broadening of the melting peak reflects a wider distribution of crystal sizes and a less perfect crystalline structure, which can be attributed to interfacial interactions between PP, PP-g-MAH, and TPS. The presence of ester linkages, as confirmed by FTIR analysis, enhances interfacial adhesion and promotes a more homogeneous dispersion of TPS, thereby influencing PP crystallization behavior.For the composite with the highest TPS content (23PP/7PP-g-MAH/70$TPS_{13/26}$), the DSC thermogram shows a further reduction in melting peak intensity and increased peak broadening. This indicates a substantial decrease in PP crystalline fraction, as the TPS-rich phase increasingly dominates the thermal response of the system. The limited continuity of the PP phase at high TPS content restricts chain folding and lamellar crystal formation, resulting in lower crystallinity and reduced melting enthalpy.

Additionally, the DSC curves of TPS-containing composites exhibit thermal transitions in the lower temperature region (approximately 100–150 °C), which can be associated with relaxation phenomena and thermal transitions within the TPS phase, including plasticizer-rich domains. These transitions become more pronounced with increasing TPS content, reflecting enhanced molecular mobility within the plasticized starch matrix.

Conclusions

In this study, thermoplastic starch (TPS) systems plasticized with different polyol compositions and their polypropylene (PP)-based composites were sys-tematically investigated in order to establish structure–property relationships and identify optimal formulations for practical applications. Based on FTIR spectro-scopic analysis, an increase in hydrogen bond density was confirmed with in-creasing plasticizer content, as evidenced by the enhanced intensity of the O–H stretching band. Simultaneously, X-ray diffraction analysis revealed the for-mation of new crystalline phases characteristic of V-type starch, including V-type and Vh-type crystals, with diffraction peaks observed at $2\theta \approx 18.3°$ and 20.8°. The highest intensities of these reflections were detected in TPS 30/00 containing 30 wt% glycerol and in TPS13/26 containing 13 wt% glycerol and 26 wt% sorbi-tol, indicating the most effective gelatinization and molecular reorganization of starch chains.On the basis of the combined FTIR and XRD results, TPS 30/00 and TPS13/26 were identified as optimal thermoplastic starch formulations for further composite preparation. These compositions exhibit a favorable balance between hydrogen bonding, molecular mobility, and crystalline organization, which is essential for obtaining structurally stable and processable thermoplastic starch.Composites prepared from polypropylene and the optimally gelatinized TPS under controlled processing conditions were subsequently evaluated in terms of their thermal and mechanical performance. Thermogravimetric and differential scanning calorimetric analyses demonstrated that the incorporation of TPS influ-ences the thermal stability and crystallization behavior of PP; however, at moder-ate TPS loadings, the composites retain acceptable thermal resistance. Mechani-cal testing revealed that the PP45/TPS50/PP-g-MAH5 composite, containing

50 wt% TPS, maintains relatively high tensile strength, stiffness, and elongation compared to composites with higher TPS contents, indicating an optimal balance between rigidity and ductility.

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