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PHYSICOCHEMICAL PROPERTIES OF THERMOPLASTIC STARCH/ HEMP BIOFILM PRODUCED BY CASTING METHOD

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Abstract. The irrational disposal of plastic waste exposes both the environment and human health to adverse consequences. This article aims to advance research in the development of new biodegradable composites based on starch and lignocellulose biomass from hemp. The objective was to develop an optimal formulation and characterize the resulting biofilm through physicochemical analyses, including FTIR to monitor changes of functional groups, TGA to assess thermal stability, and SEM to investigate morphology and changes during the introduction of filler and its distribution. Biomass filler was introduced into the polymer matrix at specified concentrations 0.1%, 0.3%, and 1%. The TGA results demonstrated comparable thermal degradation of the samples, with the primary stage of decomposition occurring at a temperature of approximately 283 °C, confirming the presence of a single basic structure of TPS in all samples. The total mass loss decreases from 93.552% to 88.552% with increasing filler concentration, thereby improving the thermal stability of the sample. SEM mass showed noticeable changes in the morphology of the samples, specifically a gradual transition from a smooth surface to more heterogeneous, fibrous structures as the filler concentration increased. The data obtained indicate that the introduction of hemp fibers increases the rigidity of the polymer matrix and improves thermal performance; however, the high filler content reduces the flexibility of the sample. In general, TPS/hemp biocomposites demonstrate high potential as biodegradable materials, making them suitable as environmentally friendly raw materials and contributing to the development of sustainable polymer biocomposites.

Key words: thermoplastic starch, corn starch, biomass, biodegradable polymer, hemp, biofilm

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ҚҰЮ ӘДІСІ АРҚЫЛЫ АЛЫНҒАН ТЕРМОПЛАСТИКАЛЫҚ КРАХМАЛ/КОНОПЛЯ БИОПЛЕНКАСЫНЫҢ ФИЗИКА-ХИМИЯЛЫҚ ҚАСИЕТТЕРІ

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Аннотация: пластикалық қалдықтарды ұтымсыз кәдеге жарату экологиялық зардаптарға да, адам денсаулығына да жағымсыз әсер әкелуі мүмкін. Бұл мақаланың мақсаты – қарасора биомассасынан крахмал мен лигноцеллюлоза негізіндегі жаңа биологиялық ыдырайтын композиттерді әзірлеу саласындағы зерттеулерді ілгерілету. Негізгі міндет алынған биомассаның оңтайлы құрамын әзірлеу және физика-химиялық талдаулар арқылы талдау, соның ішінде биокомпозиттердегі функционалдық топтардың өзгеруін бақылау үшін ИҚ-Фурье спектрометриясы, термиялық тұрақтылықты бағалау үшін ТГА және толтырғышты енгізу және оны енгізу кезіндегі морфологиялық өзгерістерді зерттеу үшін СЭМ әдісі қолданылды. Полимер матрицасына биомасса толтырғышын көрсетілген концентрацияларда енгізілді 0,1%, 0,3% және 1%. ТГА нәтижелері үлгілердің салыстырмалы термиялық ыдырауын көрсетті, ыдыраудың бастапқы кезеңі шамамен 283°С температурада болды, бұл барлық үлгілерде ТПК (термопластикалық крахмал) түрінде бірыңғай базалық құрылымның болуын растайды. Жалпы массаның төмендеуі толтырғыш концентрациясының жоғарылауы нәтижесінде 93,552% - дан 88,552% - ға дейін төмендейді, бұл өзгеріс үлгінің термиялық тұрақтылығын жақсартады. СЭМ талдауы үлгілердің морфологиясында айтарлықтай өзгерістерді көрсетті, атап айтқанда толтырғыш концентрациясы жоғарылаған сайын тегіс бетінен біртекті емес талшықты құрылымдарға біртіндеп ауысуы байқалды. Нәтижелер негізінде келесі тұжырымдамалар алынды: қарасора талшықтарын енгізу полимер матрицасының қаттылығын арттырады және температураға тұрақтылығын жақсартады; дегенмен, толтырғыштың жоғары мөлшері үлгінің икемділігін төмендететінін атап өткен жөн. Тұтастай алғанда, ТПК/қарасора (hemp) биокомпозиттері биологиялық ыдырайтын материалдар ретінде жоғары әлеуетті көрсетеді, бұл оларды экологиялық таза шикізат ретінде жарамды етеді және тұрақты полимерлі биокомпозиттердің дамуына ықпал етеді.

Түйін сөздер: термопластикалық крахмал, жүгері крахмалы, биомасса, биологиялық ыдырайтын полимер, қарасора, биопленка

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ФИЗИКО-ХИМИЧЕСКИЕ СВОЙСТВА ТЕРМОПЛАСТИЧНОЙ БИОПЛЕНКИ ИЗ КРАХМАЛА/КОНОПЛИ, ПОЛУЧЕННОЙ МЕТОДОМ ЛИТЬЯ

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Аннотация: Нерациональная утилизация пластиковых отходов чревата как экологическими последствиями, так и неблагоприятным воздействием на здоровье человека. Целью данной статьи является продвижение исследований в области разработки новых биоразлагаемых композитов на основе крахмала и лигноцеллюлозы из биомассы конопли. Основной задачей было разработать оптимальный состав и охарактеризовать полученную биоплёнку с помощью физико-химических анализов, включая ИК-Фурье-спектроскопию для мониторинга изменений функциональных групп в биокомпозитах, ТГА для оценки термостабильности и СЭМ для изучения морфологии и изменений при введении наполнителя и его распределении. В полимерную матрицу был введён наполнитель из биомассы в концентрациях 0,1%, 0,3% и 1%. Результаты ТГА продемонстрировали сопоставимый характер термической деструкции образцов: первичная стадия разложения происходила при температуре приблизительно 283 °C, что подтверждает наличие единой базовой структуры ТПК во всех образцах. Общая потеря массы уменьшается с 93,552% до 88,552% с увеличением концентрации наполнителя, тем самым повышая термостабильность образцов. СЭМ-анализ показал заметные изменения в морфологии образцов, в частности постепенный переход от гладкой поверхности к более неоднородным волокнистым структурам по мере увеличения концентрации наполнителя. Полученные данные свидетельствуют о том, что введение волокон конопли повышает жёсткость полимерной матрицы и улучшает её тепловые характеристики. Однако высокое содержание наполнителя снижает гибкость образца. В целом, биокомпозиты ТПК/конопля (hemp) демонстрируют высокий потенциал в качестве биоразлагаемых материалов, что делает их пригодными в качестве экологически безопасного сырья и способствует разработке устойчивых полимерных биокомпозитов.

Ключевые слова: термопластичный крахмал, кукурузный крахмал, биомасса, биоразлагаемый полимер, конопля, биопленка

Introduction. The global accumulation of plastic pollution poses a serious threat to the environment, characterized by "poor reversibility." Due to the slow pace of natural mineralization and the limited availability of effective recovery methods, the adverse effects of this pollution, including changes in biogeochemical cycles and damage to ecosystems, are becoming almost irreversible.

Every year, an estimated 9 to 23 million tons of plastic waste are discharged into the water environment (MacLeod, 2021). It is predicted that by 2025, these predicted emission rates will have doubled. The variety of plastic sources and types, their resistance to decomposition, and the potential harm they pose to living things have all been emphasized in numerous studies. Scientists have studied ”microplastics” in detail, specifically those categorized by size. Other plastics that have received less attention include *macroplastics* (*size > 5 mm) and nanoplastics (size < 1 µm*). Any plastic released into the environment is considered plastic pollution (Li, 2021). In addition, microplastics not only pollute the environment but also pose an invisible threat, as they can penetrate the human body through professional activities and other sources. Recent studies using Fourier-transform infrared spectroscopy (FTIR), gas chromatography, and mass spectrometry have identified nano- and microplastics in the lungs (Jenner, 2022), intestines (Santini, 2024), and even in the human placenta (Garcia, 2024). These particles include polyethylene, rayon, polypropylene, polystyrene, polyethylene terephthalate, and other unclassified plastics. Vasse G.F. et al. (2024) focused on researching how micro- and nanoplastics (MNP) pollution relate to specific lung cells and respiratory diseases such as asthma, COPD, lung cancer, and interstitial lung diseases (ILDs). According to research based on industrial and scientific data sources, a total of 10,000 substances have been identified, with more than 2,400 classified as substances of potential danger. SciFinder reports that 1327 of these substances are improperly regulated in some countries and are even connected to food (901 substances), while 266 of these substances have not been thoroughly studied (Wiesinger, 2021). In the face of increasing plastic pollution, there is a growing demand for a more environmentally friendly alternative. Creating conditions for the creation, processing, and composting of biodegradable plastic materials may be the best solution to the above problems (Flury et al., 2021), since disposal of traditional plastic (Alabi et al., 2019) (especially incineration (Nagyet al., 2016) is not the best solution because of its negative impact on the environment. The use of lignocellulosic biomass as a filler attracts the attention of researchers and has been successfully applied in various fields, including additive manufacturing, environmental remediation, and medicine. It can also be an alternative to traditional plastics (Mujtaba et al., 2023). This research aims to develop formulations of biocomposites that can serve as a solution to the aforementioned problems. The aim of the study is to study the effect of adding hemp as a reinforcing material, as lignocellulose biomass to the basic polymer matrix in the form of TPS. In the modern scientific environment, thermoplastic starch plays a crucial role as a fundamental component in the polymer matrix for the manufacture of biodegradable materials of various origins, including bioplastics (Jayarathna et al., 2022). It should be noted that starch is not used in its native form to produce starch-containing biocomposites on an industrial scale due to its disadvantages, such as poor solubility, retrogradation, syneresis, thermal decomposition, and high viscosity after gelatinization (Compart et al., 2023). To eliminate the disadvantages, starch undergoes various modifications; in our case, plasticization was used as a method of chemical modification. In granular form, starch mainly consists of linear amylose and highly branched amylopectin. When mixed with a limited amount of water, plasticizers (such as sorbitol and glycerin) are added, and starch is exposed to heat and shear forces. As a result, it spontaneously decomposes, forming a thermoplastic starch (TPS) mixture (Yu et al., 2021). Although it can be used as an environmentally friendly polymer, TPS has a hydrophilic property that makes it difficult to use directly as a biofilm. During operation, sensitivity to water leads to swelling of the material. Moreover, poor barrier and mechanical properties, such as low tensile strength and elongation at break, limit its wide application and competition with traditional film representations (Rahardiyan et al., 2023). One method to mitigate these disadvantages involves developing a biofilm formulation that incorporates lignocellulose fibers into the TPS polymer matrix. In this regard, it is crucial to consider the role of material compatibility, which is enhanced by the presence of hydroxyl groups in both starch and lignocellulose fibers, thereby forming strong interphase hydrogen bonds that improve mechanical properties (Nazrin et al., 2025). Foret et al., (2023) successfully obtained bio-composites of thermoplastic wheat starch reinforced with hemp. When studying the effect of fillers, changes in mechanical properties were observed. It was noted that as the filler concentration increases, the tensile strength and stiffness increase accordingly, which determined the threshold concentration of cannabis to be no more than 40%. There was also a decrease in water absorption as the concentration of cannabis increased. Thus, the use of cannabis as a filler has prospects as an industrial production of biofilms, contributing to a reduction in the cost of the target product. Consequently, these studies formed the basis for the production and characterization of thermoplastic starch from corn, incorporating hemp fibers into a new formulation.

Methods and materials. For this study, “Tre Mulini” corn starch (a brand from the Eurospin supermarket chain in Italy) was used as a raw material. The hemp samples were mechanically ground using a MM 400 vibratory mill (Retsch GmbH, Germany). The grinding was performed at a frequency of 100 Hz for 15 minutes until a uniform powder was achieved. Functional groups and their interactions were identified through Fourier Transform Infrared (FTIR) analysis in the range of 4000-400 cm^{-1} with a PerkinElmer, USA FT-IR Spectrometer Spectrum Two. Before the SEM analysis, the Q150T ES Plus system (Quorum Technologies, Great Britain), a device that combines spray coating and carbon evaporation, was used to enhance conductivity and prevent charging of the sample surface. The morphology and fiber-matrix interface of the composites, as well as the surface of the sample, were studied using a scanning electron microscope (SEM) with a ZEISS Sigma (FE-SEM, Carl Zeiss, Germany) system. The thermal stability and decomposition patterns of the samples were studied in an inert nitrogen atmosphere using a Discovery TGA 55 thermogravimetric analyzer (TA Instruments, USA). Thermoplastic starch (TPS) has poor mechanical and thermal properties due to its high hydrophilicity. To enhance performance, natural fibers (biomass) were often used as reinforcement in the polymer matrix. Lignocellulosic biomass from the hemp stem, obtained in Italy, was used as a reinforcement agent to improve mechanical properties. Glycerol is used as a plasticizer because of its compatibility with amylose, along with acetic acid, which acts as a co-plasticizer and significantly enhances the water absorption of the films. Staker J. et al. (2024) revealed that acetic acid possesses the property of cleaving glycosidic bonds; moreover, as a co-plasticizer with glycerin, it exhibits a high tensile strength ($M = 2.04 \pm 1.24$ MPa). The synthesis of biodegradable plastic made from starch, glycerol, and vinegar (acetic acid) was performed using the method by Abdel Hamid E.M. et al. (2025), with the formulation shown in Table 1. Each sample contains the same amount of starch, glycerol, and vinegar as indicated in Table 1 (S/G-V); however, different concentrations of hemp fiber were added (0.1%, 0.3%, 1%). Pre-weighed 15 g of corn starch was placed in a laboratory beaker and stirred with a magnetic stirrer heated to 100°C. 125 ml of distilled water was gradually added, stirring constantly with a glass rod until a homogeneous suspension was formed.

To prevent lumps from forming, the mixture was continuously stirred. Plasticizers were added sequentially, including 15 grams of glycerol and a 6% acetic acid solution. Maintaining a consistent temperature is crucial, as raising the temperature of the polymeric compound above 120°C can affect its structural integrity and textural properties. The mixture was subjected to continuous agitation until a viscous mass was established. Consequently, samples were prepared with varying concentrations of hemp fiber, which had been previously dried and milled into a powdery state. The formation of the thermoplastic film was carried out using the casting method onto a Teflon surface. Each sample was assigned a name and numbered (A1, A2, etc.). The next day, the films were pressed from above with a glass surface to keep their shape and ensure even drying. After 5-6 days, the biofilms could be carefully removed using a laboratory spatula.

Table 1 – Comparative table of TPS/hemp sample compositions

<table>
<tr><th>Sample</th><th>The ratio of components, g</th><th>Fiber (hemp powder) ratio g, %</th></tr>
<tr><td>A</td><td rowspan="4">15 g Corn starch
125 ml Demineralized water
15 g Glycerol
15 ml Vinegar (6% of acetic acid in water)</td><td>No fiber</td></tr>
<tr><td>C</td><td>Hemp fiber 0,1% (0,16 g)</td></tr>
<tr><td>D</td><td>Hemp fiber 0,3% (0,45 g)</td></tr>
<tr><td>F</td><td>Hemp fiber 1% (1,6 g)</td></tr>
</table>

Figure 1.1

Figure 1.2

Figure 1.3

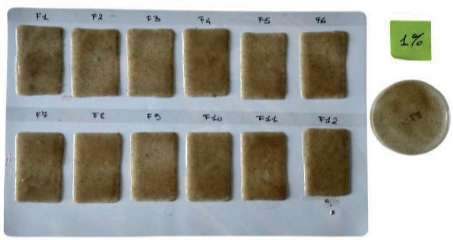


Figure 1.4

Figure 1. Samples of TPS with different concentrations of hemp fiber. TPS with 0% of hemp fiber (1.1), TPS with 0.1% of hemp fiber (1.2), TPS with 0.3% of hemp fiber (1.3), TPS with 1% of hemp fiber (1.4)

Results. Four representative samples were selected from the obtained films, one from each batch (A1, C12, D7, F11), to assess further the impact of the morphological state of the lignocellulose filler on the properties of TPS.

FTIR Analysis

The FTIR spectra for all samples have characteristic absorption bands typical of starch-containing thermoplastic materials (Figure 2). A broad and intense band is observed in the region of 3300 cm^{-1}, indicating fl uctuations in the tension of the O-H hydroxyl groups present, indicating the presence of hydroxyl groups commonly present in cellulose, hemicellulose, and lignin, as well as in adsorbed water. This breadth suggests the presence of extensive hydrogen bonding in the material. The peak value of approximately 2920 cm^{-1} corresponds to C-H stretching fl uctuations, usually associated with aliphatic methyl and methylene groups present in organic compounds. The band with an angle of 1640 cm^{-1} can be explained as corresponding to the vibrations of water molecules absorbed by cellulose.

In some cases, this may also indicate stretching of C=O from carbonyl or carboxyl groups, although its intensity and width often more strongly suggest the water content in lignocellulosic materials. The band at 1420 cm^{-1} is associated with fl uctuations of C=C Groups in aromatic compounds, and this is often indicative of the crystalline structure of cellulose. The bands at frequencies 1150 cm^{-1} and 1030 cm^{-1} of asymmetric C-O-C stretching and C-O stretching vibrations are characteristic of the intermolecular valence vibration of carbohydrates. These bands confi rm the matrix of polysaccharide structures.

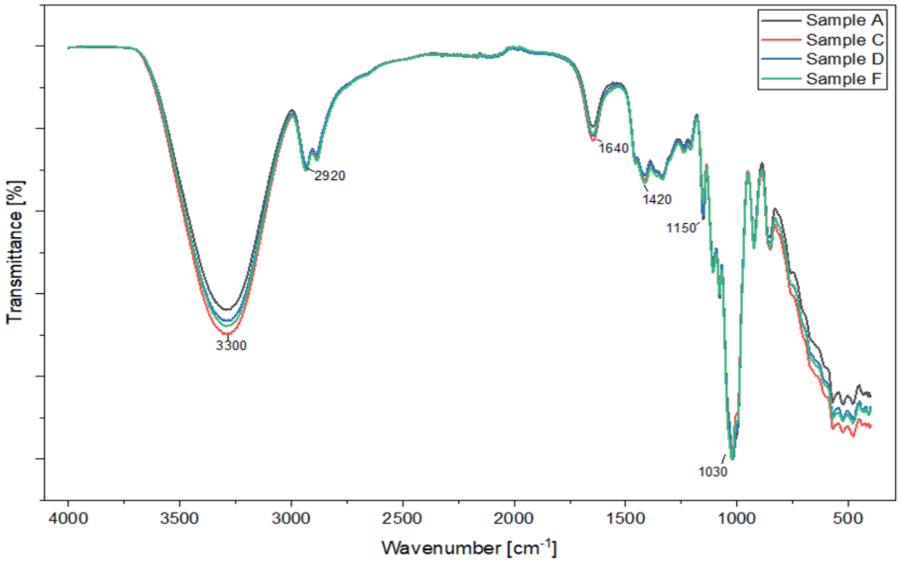


Figure 2. Comparative FTIR spectra of samples A, C, D, and F

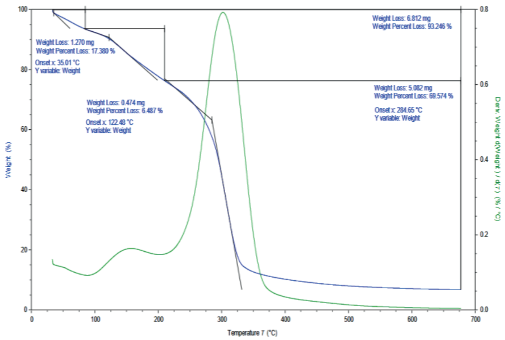
TGA Analysis. At a temperature of approximately 30.78°C, the initial mass loss of sample A was 11.508%, which is attributed to the evaporation of moisture. The second stage of degradation begins at a temperature of 143.30 °C, resulting in a further 23.871%

mass loss. This is followed by a more significant mass loss (58.344%) at a temperature of 283.90°C, which is associated with the primary thermal decomposition of TPS. The total mass loss reaches 93.552%, which means almost complete decomposition at temperatures closer to 700°C.

The initial mass loss of sample C was 17.380% at 35.01°C, followed by a slight mass loss of 6.487% at 122.48°C. At the third stage, the mass loss was 69.574% at a temperature of 284.65°C. The total mass loss was 93.246%.

Sample D exhibits an initial mass loss of 21.230% at 40.81°C, an intermediate mass loss of 13.978% at 193.89°C, and a significant degradation stage, characterized by a mass loss of 56.017% at 283.70°C. The total mass loss is 90.889%.

Sample F shows an initial mass loss of 8.402% at 46.88°C, followed by 27.568% at 166.54°C and a significant mass loss of 52.440% at 282.54°C. The total mass loss was 88,552%.



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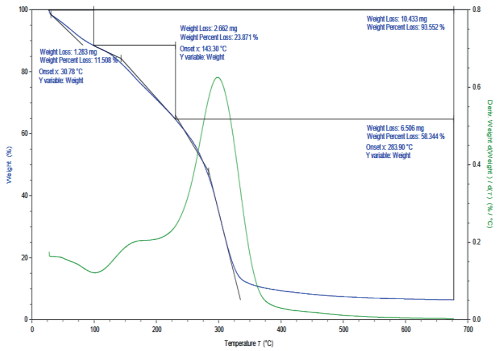

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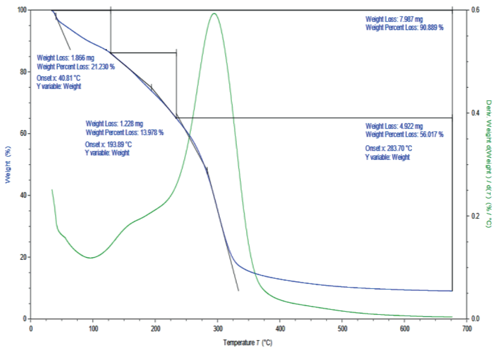

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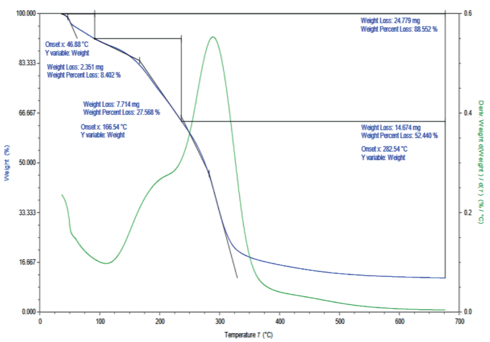
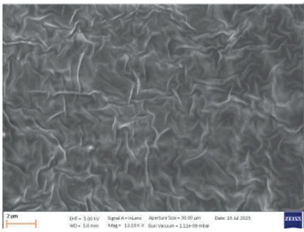


Figure 3.4

Figure 3. TGA curves for TPS with samples. Sample A (3.1), Sample C (3.2), Sample D (3.3), Sample F (3.4)

SEM Analysis. The TPS sample (A) without filler exhibits a homogeneous, wrinkled surface. This complex folded structure suggests a large surface area, typical of a pure TPS polymer matrix. Additionally, Sample C shows a more heterogeneous surface than pure TPS, characterized by irregularities and distinct aggregates. These features likely result from the uneven distribution of hemp filler within the biofilm matrix. Figure 4 displays a noticeable round, textured formation, possibly an agglomerated filler particle or a concentrated filler area. Other regions contain numerous uneven aggregates, indicating different levels of dispersion. In sample D, a distinct structural protrusion with layered features is visible, which may be a larger fiber material and has a smoother surface compared to sample C, but with more prominent particles or particles at the surface level. Sample F has complex topographic features, including highly layered, porous, and fibrous structures, as well as a shaped surface with many small spherical particles. At an increase of 29.23 KX, layered structures measuring 582.3 nm become visible, indicating the fibrous nature of the filler and allowing for the quantification of the size of individual fibrous elements.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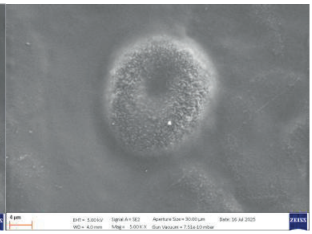
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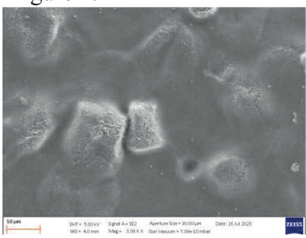
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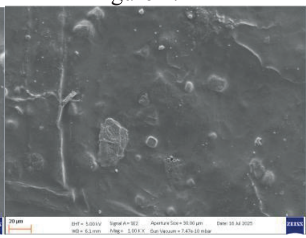
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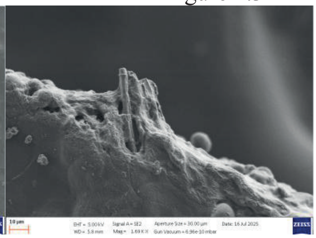
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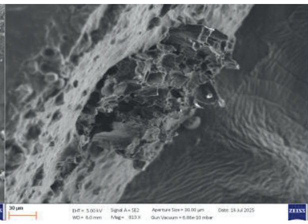
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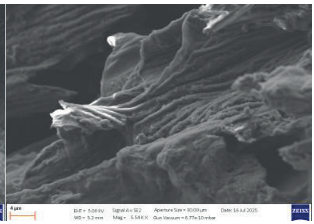
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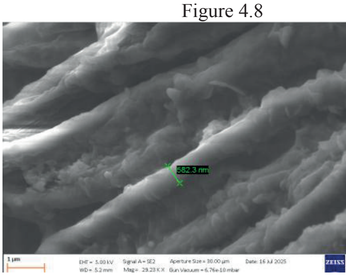

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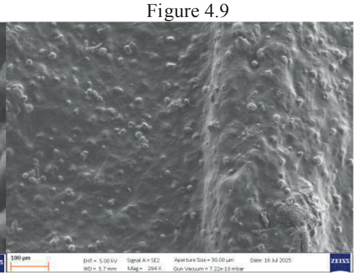
Figure 4.11

Figure 4. SEM images. Sample A (4.1, 4.2), Sample C (4.3, 4.4), Sample D (4.5, 4.6, 4.7), Sample F (4.8, 4.9, 4.10, 4.11)

Discussion. The similarity of the leading bands in the TPS matrix and the lignocellulose filler in the form of hemp does not reveal new peaks when compared. Although the overall spectral characteristics are identical for all samples due to the slight difference in the concentration of hemp fillers, minor differences in peak intensities may indicate differences in composition or structural arrangement. A modification in the intensity detected within the absorption bands at 3300 cm^{-1} indicates a differential extent of hydrogen bonding, wherein an increase in these bands is associated with a heightened concentration of the filler material. Nonetheless, it is noteworthy that the intensity of the C sample surpasses that of the samples exhibiting a greater concentration of D and F. Consequently, this variation can be elucidated by the utilization of a casting technique during the film formation process, which may facilitate an uneven distribution of the hemp fiber. The observed disparity in the band area (less than 1,500 cm^{-1}) may signify variations in the relative proportions of cellulose, hemicellulose, or lignin, as well as alterations in their crystallinity or amorphous content.

All specimens exhibit the preliminary phase of weight reduction at comparatively low thermal conditions (30-47 °C). These initial losses are commonly attributed to the drying process of absorbed fluids or the release of volatile substances present in the samples. The scale of these early losses depends on the exact sample, consequently illustrating discrepancies in their hygroscopic traits or volatile substance levels. Each specimen presents one or two intermediary phases of decomposition, occurring at approximately 120 °C to 200 °C. These moments likely indicate the erosion of less durable factors or minor structural components within the material. In all specimens, the initial temperature consistently oscillates between 282 and 285 °C, at which point a discernible stage of degradation is evident. This significant weight reduction, ranging from 52% to 69% of the total mass, signifies the primary thermal decomposition of polymeric constituents. The agreement of the primary temperature points to the availability of a shared base material, a thermoplastic starch matrix, among all samples. After the principal decomposition, the residual mass of all specimens at a temperature of 700 °C varies between approximately 6.4% and 11.4%. This residual matter may comprise inorganic fillers, carbonization, or thermally stable components that remain intact under the prescribed experimental conditions.

SEM results reveal different surface morphologies among the samples. Some images display a relatively smooth, continuous, film-like structure with some wrinkles and creases, indicating plasticization of the material. Others show rougher, more uneven surfaces with characteristic features such as round structures with granular textures and areas containing visible pores or fibrous structures.

Conclusion. The SEM investigation skillfully highlights the singular morphological characteristics of pure TPS, as well as those of the hemp filler. Figure 4 explains the evolution from a smooth, uniform polymeric structure to increasingly complex and assorted configurations as filler is added. Examining the microstructural traits is essential for understanding the consequences of filler integration on the properties of the composite, which encompass mechanical robustness, barrier capabilities, and decomposition tendencies, all of which are intimately tied to the filler's distribution, shape, and engagement within the polymer matrix. The FTIR analysis substantiates the constancy of the chemical foundation of TPS across all samples, indicating that hemp fiber predominantly functions as a filler without substantial alterations to the principal functional groups. In every examined sample, an observable initial mass reduction occurs, succeeded by a transitional phase linked to the degradation of hemp, leading to a notable stage of destruction related to the TPS structure. A large quantity of hemp, as showcased by sample F, could potentially improve hardness and strength; however, a clear decrease in flexibility is apparent. Utilizing natural hemp fibers enhances the rate at which TPS composites break down, thereby supporting the development of more environmentally sustainable materials.

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