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Doctor Blade Casting of Thin Films Containing Different Concentrated Endemic Plant Extracts: Determination of Structure and Optical Properties

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Abstract

This study provides a thorough properties of the optical analysis of the thin films which produced from *Astragalus tokatensis* Fisch., *Helichrysum noeanum* Boiss. and *Stachys huber-morathii* R. Bhattacharjee extracts. Methanol extracts of plants were obtained via Soxhlet extractor. The highest extract yield (10.10%) was determined in *H. noeanum*. Doctor blade coating method is used to make thin film layer on glass substrate. The optical behavior of the deposited films is tested by means of he UV-vis-near IR absorbance and transmittance characterization. It is found that the maximum transmittance spectra reaches nearly to a value of 90 % for *A. tokatensis* sample. Significantly, all the samples display same optical absorbace spectra behavior. Energy band gaps of the films are presented based on Tauc relation and were found to be in the range between 3.68-3.81 eV. Besides, the analysis of functional groups available in the materials is broadly studied by Fourier transform infrared (FT-IR) spectroscopy. FT-IR measurement also confirms that all produced films have carbohydrate pattern. These findings demonstrate a cost-efficient approach for the production of thin films with plant extraction, and open a new perspective on the potential applications of optoelectronic devices.

Key Words: Thin film, Optical properties, FT-IR, Energy band gap

Farklı Konsantrasyonlardaki Endemik Bitki Ekstraktlarını İçeren İnce Filmlerin Doktor Blade Yöntemiyle Üretilmesi: Yapı ve Optik Özelliklerinin Belirlenmesi

Öz

Bu çalışma *Astragalus tokatensis* Fisch., *Helichrysum noeanum* Boiss. ve *Stachys huber-morathii* R. Bhattacharjee ekstraktlarından elde edilen ince filmlerin optik analizinin kapsamlı bir araştırmasını sunmaktadır. Bitkilerin metanol ekstraktları Soxhlet ekstraktörü aracılığı ile elde edilmiştir. En yüksek ekstrakt verimi (%10,10) H. noeanum'da belirlenmiştir. Cam yüzeyine ince filmlerin üretilmesi için doktor blade yöntemi kullanılmıştır. Üretilen ince filmlerin optik davranışları UV-görünür yakın kızıl ötesi aracılığıyla soğurma ve geçirgenlik spektrumları elde edilmiştir. *A. tokatensis* örneği için maksimum geçirgenlik spekturum değerinin hemen hemen %90'a ulaştığı görülmektedir. Üretilen tüm filmler, optik soğurma spektrumunda benzer davranışlar sergilemektedir. Tauc teoremine göre, üretilen filmlerin yasak enerji bant aralıkları 3,68-3,81 eV olarak bulunmuştur. Ayrıca, malzemelerde bulunan fonksiyonel grupları analiz etmek için Fourier dönüşümlü kızıl ötesi (FT-IR) spektroskopisi kullanılmıştır. FT-IR ölçümü neticesinde üretilen filmlerin karbonhidrat modeline sahip olduğu belirlenmiştir. Bu bulgular, bitki ekstraksiyonu ile ince filmlerin üretimi için ekonomik bir yaklaşım modellemekte ve optoelektronik cihaz uygulamalarında yeni bir bakış açısı sunmaktadır.

Anahtar Kelimeler: İnce film, Optik özellikler, FT-IR, Enerji bant aralığı

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1. INTRODUCTION

Optical absorbance is a characteristic that measures the amount of light absorbed by a substance. The optical absorbance of a substance indicates how much light at a specific wavelength has been absorbed by the material (Rusak et al. 2010). Transmittance, on the other hand, is a characteristic that measures the amount of light transmitted through a substance. The transmittance of a substance shows how much light at a specific wavelength has passed through the material. These terms are commonly used in techniques like spectrophotometry (Panwar et al. 2023; Gu et al. 2024).

Optical absorbance and transmittance properties are directly related to the physiology and biochemical processes of plants (Saleque et al. 2022; Radotić et al. 2023). Plants are organisms known for their ability to absorb light, undergo photosynthesis, and utilize energy in various biological processes. In photosynthesis, plants absorb light and convert it into chemical energy (Chen et al. 2015; Saewan and Jimtaisong 2015). The pigment chlorophyll, especially, absorbs blue and red light, using this light energy in photosynthetic reactions. The optical absorbance spectrum of plants resembles the absorption spectrum of chlorophyll, indicating that plants strongly absorb light at specific wavelengths. Plant tissues, especially leaves, allow a portion of light to pass through (Barber 2003; Kiang et al. 2007). This transmitted light participates in different biochemical processes within the plant cells. Transmittance measures the ability of plant tissues to allow light to pass through. Optical measurements are commonly used to assess plant health and understand growth conditions (Muñoz-Huerta et al. 2013).

The optical absorbance or transmittance of plant leaves can provide information about plant health, stress conditions, nutrient deficiencies, and environmental factors. Plants exhibit various biological processes in response to light. Phototropism, for example, involves the directional growth of plants toward light. The optical properties of light are associated with signals that direct the growth and development processes of plants (Boichenko 2004). These points highlight the importance of understanding the optical absorbance and transmittance properties of plants to comprehend their impact on energy intake, photosynthesis, and various biological processes (Tang et al. 2020; He et al. 2021). These properties can find applications in various engineering fields. Optical properties can offer insights into plant health and growth conditions. The optical absorbance spectrum of plants can be used for early detection of factors such as diseases, pests, or nutrient deficiencies. This information can assist agricultural engineers and farmers in optimizing plant care. Efforts in engineering aim to enhance the efficiency of photosynthesis by enabling plants to use light more effectively. Optical measurements can be utilized to assess and optimize the effectiveness of such efforts (Vollmann and Eynck 2015; Sargent et al. 2022).

Optical properties are crucial for light management in indoor plant cultivation systems, such as greenhouses and vertical farming. Materials with high transmittance and suitable absorbance properties can be used to optimize growth conditions (Maraveas 2019). The optical properties of plants can be employed in environmental monitoring applications. For instance, optical measurements can track changes in plant cover and assess plant species used in environmental restoration projects. The optical properties of plants can be valuable in climate change studies, as changes in plant cover can impact the atmospheric carbon cycle (Sharma et al. 2023; Shin et al. 2023). Optical measurements can be used to understand and model such changes. Furthermore, the optical properties of plants can contribute to the design and development of plant-based sensor technologies. Sensors monitoring the optical properties of plants can guide automatic irrigation systems in agricultural fields (Omia et al. 2023).

Thin film deposition from plants is typically achieved by applying plant-based products onto a substrate. This process often involves spreading or spraying oil onto a surface. Thin film deposition is a crucial process used extensively in the food industry, pharmaceuticals, and the cosmetic industry (Wang et al. 2021a). In the food industry, thin film deposition is commonly employed for packaging materials and food preservation purposes (Shin et al. 2023). In the cosmetic industry, thin film deposition can be applied to skincare products and makeup. In the pharmaceutical industry, it is essential for coating and protecting medications. Thin film deposition offers various advantages, including material protection, product durability, and aesthetic enhancements, making it widely applicable (Chang et al. 2015; Wang et al. 2021b). Based on these properties of the plants, we investigated the optical absorbance properties of *Astragalus tokatensis* Fisch., *Helichrysum noeanum* Boiss. and *Stachys huber-morathii* R. Bhattacharjee, which have not been researched on their optical characteristics before.

2. MATERIAL AND METHOD

2.1. Collection and identification of the plant samples

Samples of *A. tokatensis*, *H. noeanum* and *S. huber-morathii* were gathered during their flowering periods from March to October in the vicinity of Kazova, located in Tokat, Turkey. The identification of plant species was carried out using various references (Davis 1988; Güner et al. 2000; Güner et al. 2012). *Astragalus tokatensis* and *H. noeanum* are classified as Irano-Turanian endemic elements, with IUCN risk categories of NT (near threatened) and LC (least concern), respectively. *Stachys huber-morathii*, a Turkey endemic, falls under the IUCN risk category of VU (vulnerable) (Tunç 2019). The entire plant samples underwent drying in an oven at 40°C until a constant weight was achieved, following the removal of soil and waste. Subsequently, the samples were ground for the purpose of the study.

2.2. Preparation of the extracts

Samples of *A. tokatensis*, *H. noeanum* and *S. huber-morathii* were air-dried and then pulverized using an ultra-centrifuge grinder (Retsch ZM 200, Germany). The ground dried plant specimens, weighing 10 grams, underwent extraction with 250 mL of methanol at room temperature utilizing a Soxhlet extraction apparatus. The resultant crude extracts from the plant samples were sieved through Whatman No. 1 filter paper. The solvent was eliminated by employing a rotary evaporator (IKA, Staufen Germany) under vacuum conditions, leading to drying, and subsequently lyophilized to obtain ultra-dry powders.

2.3. Preparation of thin films

Thin films were produced to investigate the optical properties of plant samples. The solutions of plant extract used in coating thin films were prepared at different concentrations of 5, 10, and 15 mg/mL. These solutions were coated on a glass substrate by the doctor-blade method (Figure 1). The substrate was fixed with tape at both ends, and the solution was coated on the surface with a glass rod. This process was repeated 5 times, and homogeneous thin films were obtained. Furthermore, thicker films were obtained by the drop-casting method by dropping 50 µm solution onto the substrate. Thus, thin films with different thicknesses were obtained using various coating methods.

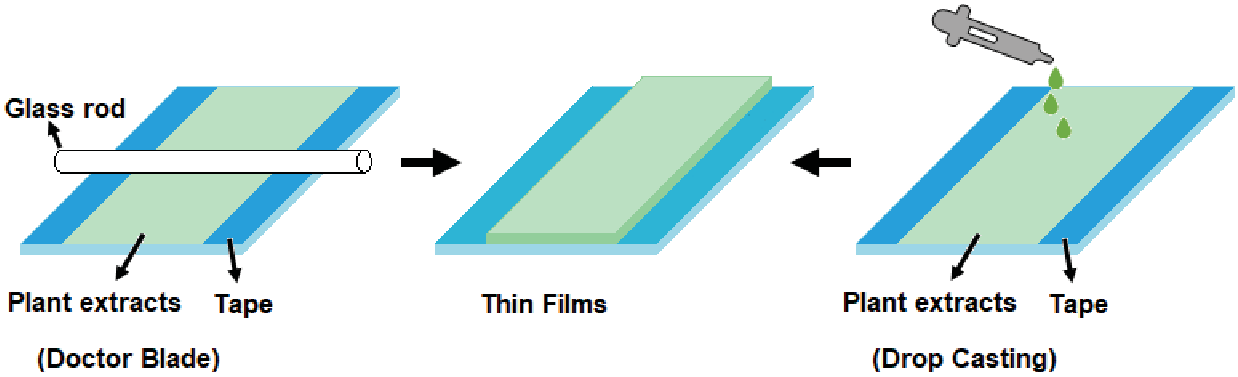

Figure 1. Graphical illustration of the thin film preparation process with Doctor Blade and drop casting method.

3. RESULTS AND DISCUSSION

The yield rates of methanol extracts obtained from *A. tokatensis, H. noeanum and S. huber-morathii* were given in Figure 2. According to yield values, extracts were in the ascending order of *A. tokatensis* (8.75%) < *S. huber-morathii* (9.00%) < *H. noeanum* (10.10%). In the heatmap analysis, extract yield rates of types *A. tokatensis* and *S. huber-morathii* showed a close gradient to each other. *H. noeanum* extract differed from other samples with a red color gradient.

The effect of sample concentration on optical properties and the band gap (Eg) values of *A. tokatensis*, *H. noeanum* and *S. huber-morathii* thin films has been investigated. The UV-vis-near IR optical absorbance and transmittance spectra of the *A. tokatensis*, *H. noeanum* and *S. huber-morathii* samples with various concentrations as a function of wavelength are displayed in Figure 3. It has been found that the intensity of absorbance increases with the concentration varied from 5 to 15 mg/mL for all the samples and it is maximum for *H. noeanum* sample. This is a result of enhancements in the particle size and aggregation of the films, contributing to a heightened light absorption. The incline in transmission occurs significantly in the vicinity of ultraviolet region because of the absorption caused by the band gap.

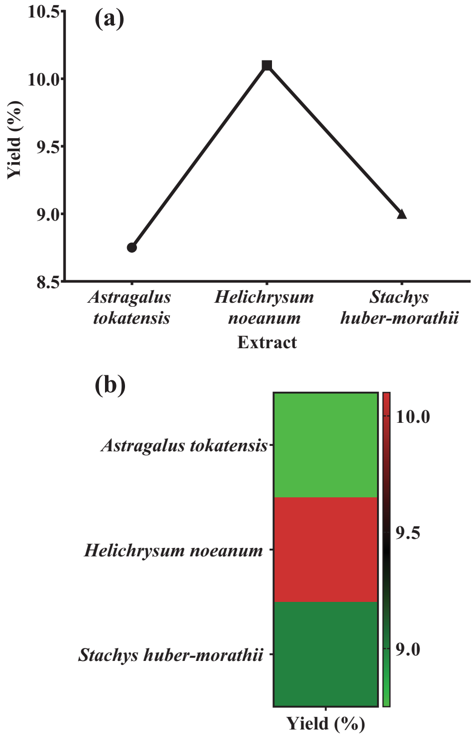

Figure 2. (a) Yield (%) of obtained the plant extracts. (b) Heatmaps depicting the percentage of yield of the methanol extracts are presented. In the heatmap analyses, high and low activities are indicated by red and green colors, respectively.

In addition, the highest average value of transmittance in the visible region (450-800 nm) varies between 69% and 90% for *A. tokatensis* sample depending on the concentration of the samples, while the lowest average value of transmittance is observed for *H. noeanum* sample.

Optical properties of our samples are comparable with the earlier literature. Zumahi et al. have reported extraction of different plant (*Portuca grandiflora*, *Rosa* ards rovar, *Celosia argentea* var. *cristia*, *Pereskia bleo*, *and Alternantera ficoidea*) extraction and then analysed aging effect at 25°C and 60°C on their optical properties (Zumahi et al. 2020). They found the transmittance in the range between nearly 60% and 95%. Also, according to their findings, the energy band gap of the samples was found to be 2.07 – 4.15 eV. Jeyaram et al. have published optical properties of chlorophyll-α extracted from *Andrographis paniculata* leaves (Jeyaram and Geethakrishnan 2019). They characterized optical properties of the sample with UV-Vis-NIR spectrophotometer and caused a similar absorbance spectrum behaviour of with our graphs. Sangeetha et al. have synthesized zinc oxide nanoparticles from *Aloe barbadensis miller* leaf extract and, the produced samples have been characterized using UV-Visible spectrophotometer (Sangeetha et al. 2011). The absorption spectra behaviour of this work is in correlation with our study in the wavelength region of 300 to 500 nm. Bajic et al. have worked on chitosan-based films with plant extracts derived from *Quercus robur, Humulus lupulus* and *Laminaris hyperborea* (Bajić et al. 2019). They found that the transmittance values varied between 33.6% and 68.7%, similarly to our *H. noeanum* sample.

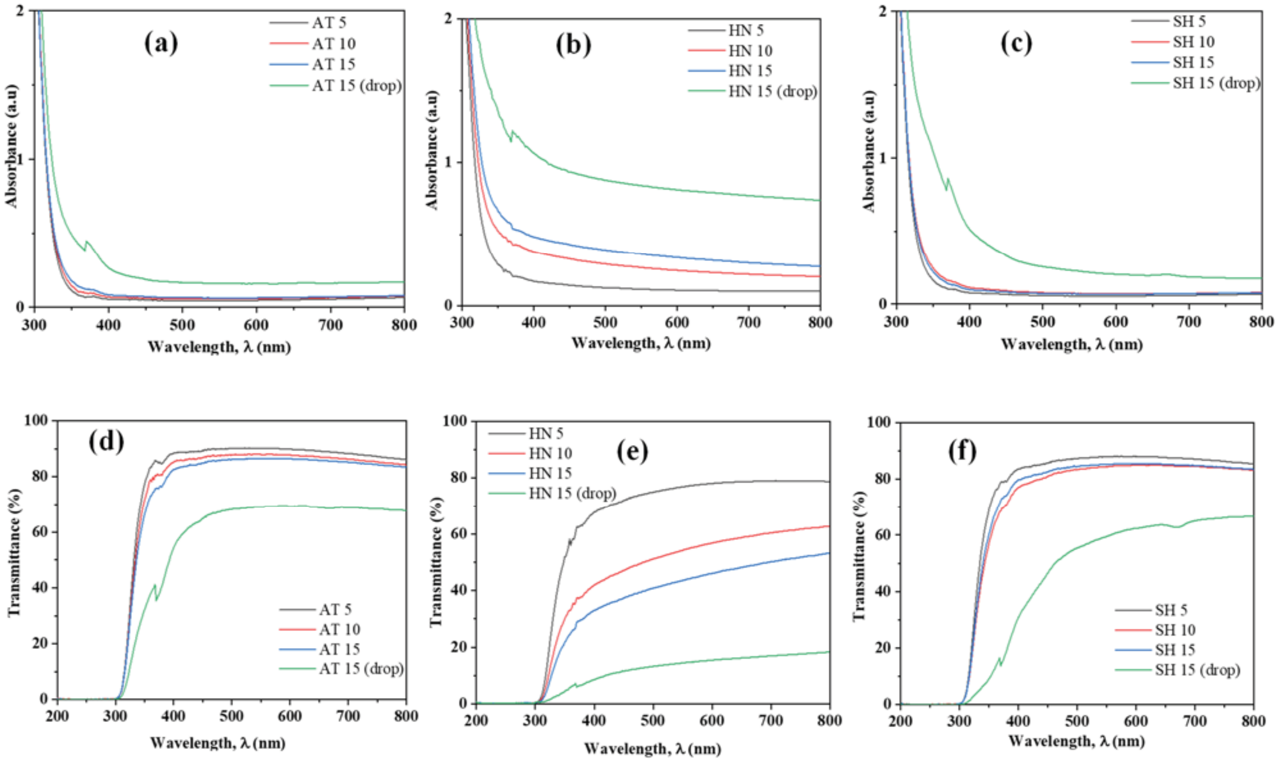

Figure 3. Optical absorbance (a-c) and transmittance (d-f) of the produced samples. AT 5, 10, 15: *A. tokatensis* extract at 5, 10 and 15 mg/mL, respectively. HN 5, 10, 15: *H. noeanum* extract at 5, 10 and 15 mg/mL, respectively. SH 5, 10, 15: *S. huber-morathii* extract at 5, 10 and 15 mg/mL, respectively.

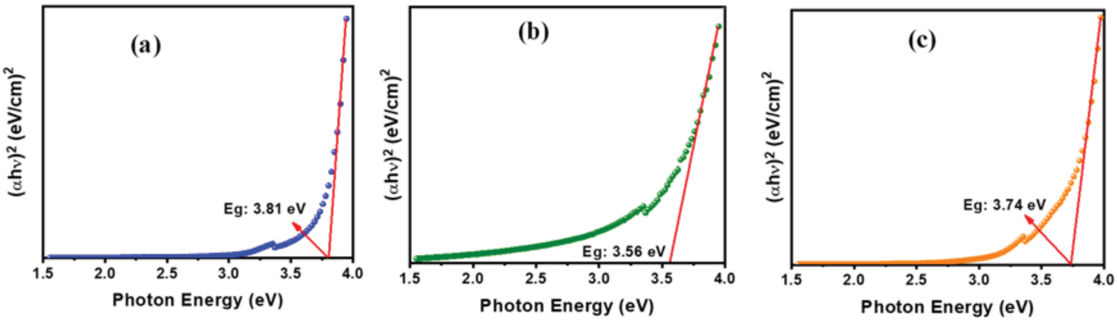

Figure 4. Tauc of the a) *A. tokatensis*, b) *H. noeanum* and c) *S. huber-morathii*

The energy band gap (E_g) of the produced samples is determined through the analysis of ultraviolet-visible (UV-vis) absorbance spectra and Tauc plots, as depicted in Figure 4, applying the following Tauc equation (Tauc et al. 1966):

$$\propto hv = A\left(hv - E_g\right)^{1/2}$$

where α is the optical absorption coefficient, hν is the incident photon energy, A is constant and E_g is the band gap energy. The optical energy band gaps for the produced samples are determined by fitting the linear parts of $(\alpha h\nu)^2$ plotted versus incident photon energy hν. The optical band gap of the samples is obtained by identifying the point where the linear portion of the intersects. From figure 4, the E_g values are 3.81, 3.56 and 3.74 eV for *A. tokatensis*, *H. noeanum* and *S. huber-morathii* samples, respectively. Consequently, it can be concluded that *A. tokatensis* samples have a higher optical energy and a higher energy and gap in comparison to the others. The determined optical values are listed in the Table 1.

Table 1. Optical parameters of the produced samples

Sample	Transmittance (at 500 nm)	Absorbance (at 500 nm)	Energy band gap (eV)
AT 5	90.08	0.045	3.81
AT 10	87.81	0.056	
AT 15	86.19	0.065	
AT 15 (drop)	68.22	0.166	
HN 5	75.01	0.125	3.56
HN 10	51.15	0.291	
HN 15	40.83	0.389	
HN 15 (drop)	13.25	0.878	
SH 5	87.3	0.059	3.74
SH 10	83.4	0.079	
SH 15	84.47	0.073	
SH 15 (drop)	55.37	0.257	

AT 5, 10, 15: *A. tokatensis* extract at 5, 10 and 15 mg/mL, respectively. HN 5, 10, 15: *H. noeanum* extract at 5, 10 and 15 mg/mL, respectively. SH 5, 10, 15: *S. huber-morathii* extract at 5, 10 and 15 mg/mL, respectively.

Significantly, the produced thin films stand out an ideal candidate for optoelectric device applications due their wide band gap energy ranging from approximately 3.0 to 4.16 eV (Narayanan and Deepak 2017; Islam and Podder 2021). Also, wide-band gap semiconductors in this band gap range can be used in high-efficiency solar cells for conversion electricity (Gloeckler and Sites 2005; McLaughlin and Pearce 2013). Moreover, these materials are suitable for the development of UV Light Emitting Diodes (LEDs) (Lu et al. 2003; Shur 2019).

FT-IR spectroscopy is commonly employed for revealing functional groups in the synthesized materials. FT-IR spectrum of the produced samples recorded within the 500 to 4000 cm^{-1} range is shown in Figure 5. All of the produced samples displayed similar characteristic FT-IR spectrum profile only with a slight change in the intensity of peaks. As can be seen from Figure 5, the depicted spectra of all samples are characteristic of carbohydrate pattern in comparison to findings reported in the existing literature (Byun et al. 2008; Jia et al. 2015). It consists of six different bands. The wide peak at 3303 cm^{-1} is assigned to the hydroxyl group of stretching vibration of O-H in alcohols and phenolic. The reason of differences in intensity can be explained by that *S. huber-morathii* sample has lower O-H groups than the others. In addition, a weak band at 2912 cm^{-1} was related to C-H stretching vibration of methyl groups (Sharifi-Rad et al. 2020). The absorption around 1375 cm^{-1} indicates the existence of the symmetrical deformation vibration of C-H (Hu et al. 2016). The absorption peak at around 1019 cm^{-1} belongs to the stretching vibrations of C-O-C and glycosidic bond. The signal at 1587 cm^{-1} corresponds to the vibration of C-O. Additionally, the appearance of a weak peak at around 1328 cm^{-1} is likely due to the C-H asymmetric stretching vibration (Zhang et al. 2015).

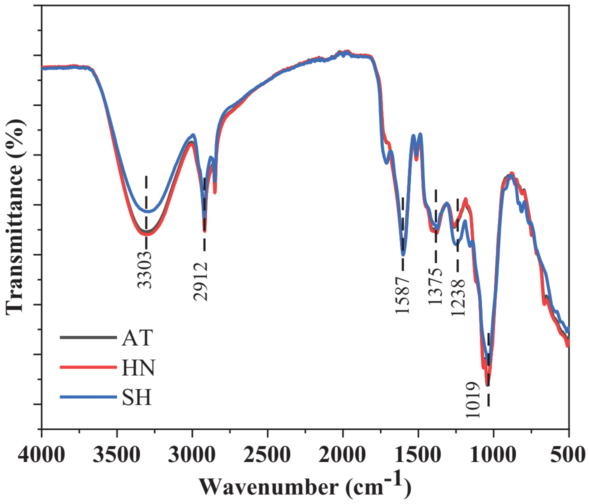

Figure 5. FT-IR spectra of the produced samples

CONCLUSION

As a conclusion, *A. tokatensis*, *H. noeanum*, and *S. huber-morathii* extracts were produced on a glass substrate using a simple doctor blade coating technique and characterized by UV-vis-near IR and FT-IR analysis. The optical investigations reveal that different concentrations of the *A. tokatensis* sample have the highest average value of transmittance of 69% and 90% in the wavelength region between 450 and 800 nm. Using Tauc relations, the energy band gap values were found to be 3.81, 3.56, and 3.74 eV for *A. tokatensis*, *H. noeanum*, and *S. huber-morathii* samples, respectively. These values are consistent with the literature on optoelectronic devices, solar cells, and UV LED applications. The remarks of FT-IR spectra showed that all the samples possess a carbohydrate pattern and no significant changes in the functional bonds between each other. Taken together, the suggested technique enables a straightforward approach for creating thin films of plant extraction.

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